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Analytical Report

PFOA and PFOS Analysis of Deer Serum Samples by LC/MS/MS

MPI Report No. L0019345

Testing Laboratory

MPI Research, Inc.
3058 Research Drive
State College, PA 16801

Requester/Project Manager

Dena Haverland
Dalton Utilities
PO BOX 869
Dalton, GA 30722
Phone: 706-529-1010

2010 JAN - 6 P 1:44

1 Introduction

Results are reported for the analysis of deer serum samples received at MPI Research from Dalton Utilities. The MPI Research study number assigned to the project is L0019345. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

| Compound Name | Acronym |
|--------------------------|----------------------|
| Perfluorooctanoic Acid | C8 Acid or PFOA |
| Perfluorooctanesulfonate | C8 Sulfonate or PFOS |

2 Sample Receipt

Two samples were received from Dena Haverland at Dalton Utilities for this study. The samples were collected on October 02, 2009. The samples arrived on October 06, 2009 via Fedex and were logged in under MPI Research login number L0019345. The shipment was received frozen on dry ice. The samples were stored frozen at approximately -80°C from receipt until analysis. Chain-of-custody information is presented in Attachment A.

3 Methods - Analytical and Preparatory

3.1 Serum Sample Preparation

- 3.1.1. Measure 1 mL of serum sample into a 50 mL disposable centrifuge tube and fortify, if appropriate. Add 0.2 mL of a 100 ng/mL WIS for a final concentration of 0.5 ng/mL.
- 3.1.2. Add water to sample for a final volume of 20 mL. Cap tightly and vortex for ~1 minute.
- 3.1.3. Transfer 1 mL of the sample using a disposable pipette into 15 mL disposable centrifuge tubes. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.
- 3.1.4. Centrifuge tubes at ~3000 rpm for ~ 5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.
- 3.1.5. Place the unconditioned SPE columns on the vacuum manifold. Condition the SPE columns by passing ~ 10 mL of methanol through the column followed by ~ 5 mL of water. The washes may be pulled through the SPE column using vacuum at a flow rate of ~1 drop/sec or may be allowed to pass through the column unaided. Discard all washes. Do not allow the column to dry.
- 3.1.6. Load the sample onto a conditioned SPE column . Discard the eluate. Any analyte residues will be trapped on the SPE column at this point.
- 3.1.7. Elute with 2 mL of methanol. Collect 2 mL of elute into a graduated 15 mL centrifuge tube.

3.2 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against external calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 4000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna 3 μ C8(2) Mercury, 20 x 4.0 mm column was used for separation.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water
Mobile Phase (B): Methanol

| Time | %A | %B |
|------|----|-----|
| 0.0 | 90 | 10 |
| 0.5 | 90 | 10 |
| 2.0 | 10 | 90 |
| 5.0 | 10 | 90 |
| 5.1 | 0 | 100 |
| 6.0 | 0 | 100 |
| 6.1 | 90 | 10 |
| 10.0 | 90 | 10 |

The following parameters were used for operation of the mass spectrometer:

| Parameter | Setting |
|-----------------------|--|
| Ionization Mode | Electrospray |
| Polarity | Negative |
| Transitions Monitored | 413→369 (PFOA) 499→80 (PFOS) 415→370 (Internal Std. ^{13}C PFOA (m+2)) 503→80 (Internal Std. ^{13}C PFOS (m+4)) |
| Gas Temperature | 450°C |

4. Analysis by LCMSMS

4.1 Calibration

For the serum sample analysis, a 6-point calibration curve was analyzed throughout the analytical sequence for PFOA and PFOS. The calibration points were prepared at 0.1, 0.2, 0.5, 1.0, 2.0, 5.0 ng/mL (ppb) each containing 1.0 ng/mL ^{13}C -PFOA (m+2) and ^{13}C -PFOS (m+4).

The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

4.2 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to deer serum controls. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

4.3 Matrix Spikes

One matrix spike was prepared by adding a known concentration of the target analyte to a sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spike was within the acceptable range with the exceptions of:

L19345-2 (Deer # 7 3.5 yr male serum) Spk C at 1000 ng/mL for PFOS, which gave low recovery of 68%.

4.4 Laboratory Duplicates

One sample was prepared in duplicate and analyzed. Duplicate results are given along with the sample results in Attachment B.

5. Data Summary

Please see Attachment B for a detailed listing of the analytical results. For the serum samples the results are reported in parts per billion (ng/mL) on an as-received basis.

6. Data/Sample Retention

Samples are disposed of 60 days after the report is issued unless otherwise specified by the project manager. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

7 Attachments

- 7.1 Attachment A: Chain of Custody
- 7.2 Attachment B: Analytical Results
- 7.3 Attachment C: Raw Analytical Data for Water

8 Signatures



Mark Neeley, Research Chemist Associate II

11-6-09
Date



Robert Zhu, Manager, Analytical

11/9/09
Date

A

Login

Login Group: L0019345

| | | | |
|---|------------------------------|---------------------|------|
| Login #: | 19459 | Conform COC Sample: | True |
| Project: | P0005195 | Conform COC: | True |
| Company Name: | Dalton Utilities | Conform Sample: | True |
| Submitted By: | Dena Haverland | Conform Request: | True |
| Login Type: | Immediate Receipt of Samples | | |
| Started: | True | | |
| Date Start: | 10/27/2009 | | |
| Due Date: | 11/06/2009 | | |
| Login Initiated: | 10/27/2009 | | |
| Received By: | Ammerman, Mark | | |
| Spread Sample: | | | |
| Label: | | | |
| MPI SD/PI: | Zhu, Xiang | | |
| Project Title/Type: PFOA and PFOS Analysis of Serum Samples By LC/MS/MS / ROUTINE | | | |
| Login Notes: | | | |

Packages / Containers

| <u>Package</u> | <u>Carton</u> | <u>Date / Condition</u> | | <u>Shipper / ID</u> | <u>Temp. Control/Temp.</u> | <u>Direction / Handled By</u> |
|--------------------|---------------------|--|-----------------------|-------------------------|----------------------------|-------------------------------|
| K0022041 | | Received Date: 10/6/09 10:25 Package & Contents Uncompromised | | FEDEX 8694 2057 8178 | Dry Ice -79.2 | RECEIVED Ammerman, Mark |
| <u>Container #</u> | <u>Gross Weight</u> | <u>pH</u> | <u>Container Type</u> | <u>Preservative</u> | <u>Mfg. Lot</u> | <u>Mfg. ID</u> |
| C0457604 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457605 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457606 | 3.20 g | | 2 ml clear plst vial | NONE | | |
| C0457607 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457608 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457609 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457610 | 3.20 g | | 2 ml clear plst vial | NONE | | |
| C0457611 | 3.40 g | | 2 ml clear plst vial | NONE | | |
| C0457612 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457613 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457614 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457615 | 3.20 g | | 2 ml clear plst vial | NONE | | |
| C0457616 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457617 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457618 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457619 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457620 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457621 | 3.10 g | | 2 ml clear plst vial | NONE | | |
| C0457622 | 3.20 g | | 2 ml clear plst vial | NONE | | |
| C0457623 | 3.10 g | | 2 ml clear plst vial | NONE | | |



Login

Samples

| <u>Sample ID</u> | <u>Container</u> | <u>Matrix</u> | <u>System</u> | <u>System Matrix</u> | <u>Sample</u> | <u>Date Sampled</u> | <u>Date Due</u> |
|------------------|------------------|---------------|---------------|----------------------|-----------------------------|---------------------|-----------------|
| L0019345-0001 | C0457604 | LIQUID | Deer | Serum | Deer #6 0.5 yr female-serum | 10/02/2009 | 11/06/2009 |
| | C0457605 | | | | | | |
| | C0457606 | | | | | | |
| | C0457607 | | | | | | |
| | C0457608 | | | | | | |
| | C0457609 | | | | | | |
| | C0457610 | | | | | | |
| | C0457611 | | | | | | |
| | C0457612 | | | | | | |
| | C0457613 | | | | | | |
| L0019345-0002 | C0457614 | LIQUID | Deer | Serum | Deer #7 3.5 yr male-serum | 10/02/2009 | 11/06/2009 |
| | C0457615 | | | | | | |
| | C0457616 | | | | | | |
| | C0457617 | | | | | | |
| | C0457618 | | | | | | |
| | C0457619 | | | | | | |
| | C0457620 | | | | | | |
| | C0457621 | | | | | | |
| | C0457622 | | | | | | |
| | C0457623 | | | | | | |

Login Reviewed By:



Date/Time:

10/28/09 1440



Sample Submittal



MPI Research Contact: Daniel Wright

Send Report To:

Company: Dalton Utilities
1200 VD Parrott JR Parkway, PO Box 869
Address:

City, State, ZIP: Dalton, GA 30722-0869

Attention: Dena Haverland

Phone #: 706-529-1010

Fax #: 706-529-1271

Email: dhaverland@dutil.com

Study/Job #:

Signature/Date:

Printed Name:

Please fax this form before sending samples.

Please send samples to shipping and receiving:
3048 Research Drive, State College, PA 16801
T: (814) 272-1039 • F: (814) 272-1019

Turnaround time (TAT) requirements:

Results Due Date: 30 days

Preliminary Results Format: Verbal Email Fax

Report Due Date: 30 days

Storage Conditions

Room temperature
Refrigerator
 Freezer
Ultra Low freezer
Desiccated
Lighting required

Stability (°C/%RH):

Stability time period:

Safety Information

Special handling:

MSDS attached

Controlled substance:

HAZARDS:

Please fill in the diamond HMIS/NFPA
(0-4) if appropriate

| Client ID# Description | Lot/ Control # | Amt. Sent/ Weight | # of Bottles | Matrix | Date & Time | Tests Requested |
|-------------------------------------|-------------------|----------------------|-----------------|--------|----------------|-----------------|
| 1 Deer #6 0.5 yr female-Serum | | 10ml | 10 | deer | 10/2/09 1:08AM | PFOA/PFOS |
| 2 Deer #6 0.5 yr female - muscle | | as requested | 1bas | deer | 10/2/09 2:28AM | PFOA/PFOS |
| 3 Deer #6 0.5 yr female - Liver | | Whole | 1bas | deer | 10/2/09 2:30AM | PFOA/PFOS |
| 4 Deer #7 3.5 yr Male - Serum | | 10ml | 10 | deer | 10/2/09 1:45AM | PFOA/PFOS |
| 5 Deer #7 3.5 yr Male - muscle | | as requested | 1bas | deer | 10/2/09 2:45AM | PFOA/PFOS |
| 6 Deer #7 3.5 yr Male - Liver | | Whole | 1bas | deer | 10/2/09 2:48AM | PFOA/PFOS |
| 7 | | | | | | |
| 8 | | | | | | |
| 9 | | | | | | |
| 10 | | | | | | |

PO #:

Notes:

| Relinquished by | Date | Time | Received by | Date | Time |
|-----------------|---------|--------|-------------|---------|------|
| Daniel Kavany | 10/5/09 | 6:30pm | | 10/6/09 | 1027 |

V00028362

Administrative Form



TEMPORARY SAMPLE STORAGE FORM

To be completed during ExyLIMS Login

Project #: P5195

Login #: L19345

Initials / Date: MJA 10/27/09

One form to be completed for each package

Date / Time Received: 10/06/09 1025

Received By: Mark Amerson

Shipper: FedEx

Shipper Package ID: 86942057 8178

Temperature (deg C) / Thermometer ID: -79.2 1D0001715

Temperature Control Method: dry ice

Temporary Storage Location: freezer 32

Condition of sample(s):

- Good – Package and contents uncompromised
- Fair – Package damaged / contents uncompromised
- Poor – Package and contents compromised

Notes:

FedEx® USAirbill

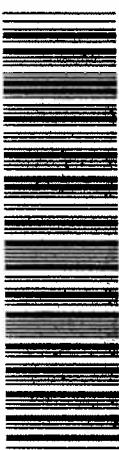
Express

FedEx
Tracking
Number

8694 2057 8178

Recipient's Copy

L



8694 2057 8178

1 From

Date

Senders Name **Daniel K. Knapp** Phone **(717) 596-5637**

Company **USPS W.H. Smith Booksellers** Address **1000 Market Street** City **Philadelphia** State **PA** ZIP **19106** ReadThisForm

2 Your Internal Billing Reference

3 To

Recipients Name

Phone **(717) 232-1029** ReadThisForm

Company **M.P.T. Booksellers Inc.** Address **3049 Broadway** City **Philadelphia** State **PA** ZIP **19104** ReadThisForm

Address **To request a package be held at a specific FedEx location, print FedEx address here:**

City **Philadelphia** State **PA** ZIP **19104**

4a Express Package Service

- FedEx Priority Overnight Next business morning. Friday shipments will be delivered on Monday unless SATURDAY Delivery is selected.
- FedEx Standard Overnight Next business afternoon. Saturday Delivery NOT available.
- FedEx First Overnight Expedited business morning delivery to select locations. Saturday Delivery NOT available.

- FedEx 2 Day Freight Second business day. Thursday shipments will be delivered on Monday unless SATURDAY Delivery is selected.
- FedEx Express Saver Same day delivery. Saturday Delivery NOT available.
- FedEx Express One Day Same day delivery. Saturday Delivery NOT available.

- FedEx 10 Day Freight Not available for FedEx Standard or Express. Second business day. Saturday Delivery NOT available.
- FedEx 30 Day Freight Third business day. Saturday Delivery NOT available.
- FedEx 60 Day Freight Fourth business day. Saturday Delivery NOT available.

* To most locations. ** To most locations.

4b Express Freight Service

- FedEx Envelope* Includes FedEx Small Pak, FedEx Large Pak and FedEx Super Pak.
- FedEx Pak* Includes FedEx Small Pak, FedEx Large Pak and FedEx Super Pak.
- FedEx Box Includes FedEx Small Pak, FedEx Large Pak and FedEx Super Pak.
- FedEx Tube Includes FedEx Small Pak, FedEx Large Pak and FedEx Super Pak.
- FedEx Other Includes FedEx Small Pak, FedEx Large Pak and FedEx Super Pak.

5 Packaging

- SATURDAY Delivery Not available for FedEx Standard or Express. FedEx First Overnight. FedEx 10 Day Freight.
- HOLD Wednesday Not available for FedEx Standard or Express. FedEx First Overnight. FedEx 10 Day Freight.
- FedEx Saturday Not available for FedEx Standard or Express. FedEx First Overnight. FedEx 10 Day Freight.

Indicate FedEx options in Section 3.

** To most locations.

6 Special Handling

Draw this shipment contains dangerous goods? No Yes One box must be checked.

As per attached Shipper's Declaration, not required.

Dangerous goods (including dry ice) cannot be shipped in FedEx packaging.

7 Payment Bill to:

Sender Recipient Third Party Credit Card Cash/Check

Enter FedEx Acct. No. or Credit Card No. below.

Obtain Recip. Acct. No. Credit Card Cash/Check

Total Packages Total Weight Total Declared Value

To request a package be held at a specific FedEx location, print FedEx address here.

Credit Card Acct.

8 Residential Delivery Signature Options

If you require a signature, check Direct or Indirect.

- No Signature Direct Signature If no one is available at home, the recipient's alternate name and address may be given or a telephone number may be given.
- Residential Delivery Indirect Signature If no one is available at home, the recipient's alternate name and address may be given or a telephone number may be given. Sign for delivery, no signature.

520

fedex.com 1800.667.fedex 1.800.463.3339

FedEx 10 Day Freight is not available in PA ZIP 19104. FedEx® PRINTED IN U.S.A.

B



3058 Research Drive
State College, Pennsylvania 16801 USA
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Fax: 814.272.1019

Analytical Report

Summary of Fluorochemical Residues in Serum Samples

| Sample ID | PFOA Perfluorooctanoic Acid | PFOS Perfluorooctanesulfonate |
|------------------------------|----------------------------------|----------------------------------|
| | Analyte Found (ng/mL, ppb) | Analyte Found (ng/mL, ppb) |
| Deer # 7 3.5 yr male-serum | NQ | 670^ |
| Deer # 7 3.5 yr male-serum* | NQ | 671^ |
| Deer # 6 0.5 yr female-serum | ND | 113 |

*Laboratory Duplicate

ND = Not detected = Response is below the LOD of 1.0 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/mL (ppb).

^ The corresponding Matrix Spike recovery was outside the acceptance criteria of 70-130%, therefore the sample values should be considered an estimate.



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Recovery Summary of Fluorochemical Residues in Serum Samples

| Sample Description | Amount Spiked (ng/mL) | PFOA | | | PFOS | | |
|--|-----------------------|-----------------------------|--------------------------|--------------|-----------------------------|--------------------------|--------------|
| | | Amt Found in Sample (ng/mL) | Amount Recovered (ng/mL) | Recovery (%) | Amt Found in Sample (ng/mL) | Amount Recovered (ng/mL) | Recovery (%) |
| LCS A (Data set 110309A) 10 ng/mL | 10 | ND | 10.1 | 101 | ND | 9.99 | 100 |
| LCS B (Data set 110309A) 50 ng/mL | 50 | ND | 47.7 | 95 | ND | 47.9 | 96 |
| LCS A (Data set 110509A) 10 ng/mL | 10 | N/A | N/A | N/A | ND | 10.5 | 105 |
| LCS B (Data set 110509A) 50 ng/mL | 50 | N/A | N/A | N/A | ND | 49.2 | 98 |
| Deer # 7 3.5 yr male-serum (L19345-2 Spk C, 50 ng/mL Lab Spike) | 50 | NQ | 51.0 | 102 | 670 | ** | ** |
| Deer # 7 3.5 yr male-serum (L19345-2 Spk C, 1000 ng/mL Lab Spike) | 1000 | N/A | N/A | N/A | 670 | 1350 | 68* |

ND = Not detected = Response is below the LOD of 1.0 ng/mL.

NQ = Not quantifiable = Response is between the LOD and the LOQ of 10 ng/mL.

* The Matrix Spike recovery was outside the acceptance criteria of 70-130%, therefore the sample values should be considered an estimate.

** The endogenous level of PFOS in the sample significantly exceeds the spiking level, therefore an accurate recovery cannot be calculated.

C

RAW DATA REPORT

| | | | | | |
|-------------------|------------|------------------------|------------|------------------|-------------------|
| Sponsor Study No: | NA | Limit of Quantitation: | 10 ng/mL | Set No: | 110309A |
| MPI Study No: | L19345 | Injection Volume: | 15 μ L | Analyst: | Mark Neely |
| Analyte: | PFOA | Matrix: | Deer Serum | Instrument Type: | LC/MS/MS Unit # 9 |
| Ions Monitored: | 413 -> 369 | | | Extraction Date: | 11/03/09 |
| Site: | NA | | | Analyzed on: | 11/03/09 |

| MPI Research ID | Sponsor ID | Sample Code | Sample Index No. | Internal | | | | Internal | | Amount | | |
|-----------------|--------------------------------------|-------------|------------------|--------------------|--------------------|---------------------|----------------------|-----------|--------------------|-----------------------|-----------------------|--------------|
| | | | | Std. Conc. (ng/mL) | Std. Conc. (ng/mL) | Aliquot Factor (AF) | Dilution Factor (DF) | Peak Area | Standard Peak Area | Analyte Found (ng/mL) | Analyte Added (ng/mL) | Recovery (%) |
| SS33618 | - | CS | 1 | 0.100 | 1.0 | - | - | 53649 | 578811 | - | - | - |
| SS33617 | - | CS | 2 | 0.200 | 1.0 | - | - | 102736 | 577969 | - | - | - |
| SS33616 | - | CS | 3 | 0.500 | 1.0 | - | - | 239879 | 580849 | - | - | - |
| SS33615 | - | CS | 4 | 1.00 | 1.0 | - | - | 496152 | 567252 | - | - | - |
| SS33614 | - | CS | 5 | 2.00 | 1.0 | - | - | 961042 | 576511 | - | - | - |
| SS33613 | - | CS | 6 | 5.00 | 1.0 | - | - | 2338317 | 556934 | - | - | - |
| Methanol Wash | - | W | 7 | - | - | - | - | 1023 | 0 | - | - | - |
| Methanol Wash | - | W | 8 | - | - | - | - | 1008 | 0 | - | - | - |
| Control | MC4311 Deer Serum Control | C | 9 | - | 0.5 | 40 | 1 | 2392 | 347101 | ND | - | - |
| LCS A | MC4311 Deer Serum Spike A | LCS | 10 | - | 0.5 | 40 | 1 | 154309 | 355719 | 10.1 | 10 | 101 |
| LCS B | MC4311 Deer Serum Spike B | LCS | 11 | - | 0.5 | 40 | 1 | 751597 | 374371 | 47.7 | 50 | 95 |
| L19345-2 Spk C | Deer # 7 3.5 yr male-serum Spike C | LF | 12 | - | 0.5 | 40 | 1 | 822293 | 383369 | 51.0 | 50 | 102 |
| L19345-2 | Deer # 7 3.5 yr male-serum | S | 13 | - | 0.5 | 40 | 1 | 18517 | 368323 | NQ | - | - |
| L19345-2 Dup | Deer # 7 3.5 yr male-serum Duplicate | S | 14 | - | 0.5 | 40 | 1 | 20592 | 412407 | NQ | - | - |
| L19345-1 | Deer # 6 0.5 yr female-serum | S | 15 | - | 0.5 | 40 | 1 | 7527 | 362334 | ND | - | - |
| SS33615 | - | CCV | 16 | 1.00 | 1.0 | - | - | 479482 | 556775 | 1.02 | 1.0 | 102 |

Analyte Found (ng/mL) = (((analyte peak area/IS peak area) - intercept) / slope) x IS conc.) x AF x DF

Standard Curve: Linear (1/x weighted)

Intercept = 0.00876

Slope = 0.838

Coef. Of Det. = 0.9998

Recovery (%) = $\frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$

CS = Calibration standard

LF = Lab fortified sample

W = Methanol Wash

CCV = Continuing Calibration Verification

FF = Field fortified sample

ND = Not detected = Response between 0 and LOD

C = Control sample

LCS = Laboratory Control Spike

NQ = Not quantifiable = Response between LOD and LOQ

Spreadsheet prepared by: Mark / 11-6-09



3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

Internal Chain of Custody/Fortification Sheet

MPI Research Study Number:
Sponsor Study/Protocol No:

L19345
NA

Matrix: Deer Serum

The samples listed below were removed from Freezer No. 37
Time 8:45 AM

Date 11-3-09

Initials MDP

| CLIENT SAMPLE ID | MPI RESEARCH ID NUMBER | VOLUME (mL) | FORTIFICATION (ng) |
|-----------------------------|------------------------|-------------|--------------------|
| na | MC4311 Control | 1.0 | - |
| na | MC4311 LCS A | 1.0 | 10 |
| na | MC4311 LCS B | 1.0 | 50 |
| Deer #7 3.5 yr male-serum | L19345-2 Spk C | 1.0 | 50 |
| Deer #7 3.5 yr male-serum | L19345-2 | 1.0 | - |
| Deer #7 3.5 yr male-serum | L19345-2 Dup | 1.0 | - |
| Deer #7 0.5 yr female-serum | L19345-1 | 1.0 | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
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| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |

| | Spiking Solution Used | Volume Used for Spiking | Initial/Date |
|----------------|------------------------|--------------------------------|-----------------------------|
| MC4311 LCS A | SS0033620 (200 ng/mL) | 50 µL (50-250µL auto-pipette) | <u>MDP</u> / <u>11-3-09</u> |
| MC4311 LCS B | SS0033619 (1000 ng/mL) | 50 µL (50-250µL auto-pipette) | <u>MDP</u> / <u>11-3-09</u> |
| L19345-2 Spk C | SS0033619 (1000 ng/mL) | 50 µL (50-250µL auto-pipette) | <u>MDP</u> / <u>11-3-09</u> |
| All Samples | SS0032652 (100 ng/mL) | 200 µL (50-250µL auto-pipette) | <u>MDP</u> / <u>11-3-09</u> |

All samples were measured using a digital autopipet IN 684.

Time 10:40 AM

Date 11-3-09

Initials MDP

After measuring samples were returned to Freezer No. 4

Time 10:44 AM

Date 11-3-09

Initials MDP

Comments:

Analysis Summary:

Data Set: 110309A

Initials/Date: MDP / 11-3-09

Data Set: 110507A

Initials/Date: MDP / 11-5-09

Data Set: -

Initials/Date: - / -

Set extraction/analysis data verified by: Amber

Date: 11/09/09

July 02, 2007/2



MPI
RESEARCH

3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

PROTOCOL NUMBER: NA

METHOD: V5821

ANALYTES: PFOA & PFOS

MPI STUDY NUMBER: L19345
MATRIX: Deer Serum

| Client ID | MPI Research ID | STEP 1 | STEP 2 | STEP 3 | STEP 4 | STEP 5 | STEP 6 | STEP 7 | Dilutions (mL/mL) | STEP 8 | Reagents/ Materials | ExyLims ID |
|-----------------------------|-----------------|---------|---------|---------|---------|---------|---------|---------|-------------------|---------|---------------------|------------|
| na | MC4311 Control | - | - | - | - | - | - | - | - | - | Acetonitrile | REY7645 |
| na | MC4311 LCS A | - | - | - | - | - | - | - | - | - | Water | REY7649 |
| na | MC4311 LCS B | - | - | - | - | - | - | - | - | - | Methanol | REY7649 |
| Deer #7 3.5 yr male-serum | L19345-2 Spk C | - | - | - | - | - | - | - | - | - | SPE Column | MA57822 |
| Deer #7 3.5 yr male-serum | L19345-2 | - | - | - | - | - | - | - | - | - | Initials/Date | MA11-3-27 |
| Deer #7 3.5 yr male-serum | L19345-2 Dup | - | - | - | - | - | - | - | - | - | - | - |
| Deer #7 0.5 yr female-serum | L19345-1 | V | V | V | V | V | V | V | V | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| - | - | - | - | - | - | - | - | - | - | - | - | - |
| *Initials/Date | - | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 | 11-3-27 |

STEP 1: Measure 1 mL of serum into a 50 mL disposable centrifuge tube and fortify if appropriate. Add 0.2 mL of internal standard (100 ng/mL).

STEP 2: Add water to the sample for a final volume of 20 mL. Cap tightly and vortex for ~1 minute.

STEP 3: Transfer 1.0 mL of the sample to a 1.5 mL centrifuge tube. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.

STEP 4: Centrifuge tubes at ~3000 for ~5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.

STEP 5: SPE Clean-up: Condition column with ~10 mL of methanol followed by ~5 mL of water at a rate of ~1 drop/second. Discard all washes. Do not allow column to go dry.

STEP 6: Load the sample onto the SPE Column. Discard the eluate. Elute with 2 mL of methanol into a 15 mL centrifuge tube. Transfer the extract into autosampler vials.

STEP 7: LC/MS/MS analysis.

STEP 8: LC/MS/MS reanalysis.

*Initials and date under each step indicates the personnel that performed this step.

COMMENTS:

Final extracts stored in refrigerator 34 Initials: MDD4 Date: 11-3-27

Oct 30, 2009/1

alyst Version: 1.4.2
rinting Time: 2:19:24 PM
rinting Date: Tuesday, November 03, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

object: \\sc1wp5556\mdrive\PE SCIEX DATA\Projects\P5195 Batch:09_110309A Serum Tab:Sample Set:SET1 AcqMethod:P5195_102
Sample

MDP 11-3-09

| | Sample Name | Sample ID | Vial Position | Data File |
|----|----------------|--|---------------|--------------------|
| 1 | SS33618 | Calibration Standard, 0.1 ng/mL | 1 | 09_110309A\110309A |
| 2 | SS33617 | Calibration Standard, 0.2 ng/mL | 2 | 09_110309A\110309A |
| | SS33616 | Calibration Standard, 0.5 ng/mL | 3 | 09_110309A\110309A |
| | SS33615 | Calibration Standard, 1.0 ng/mL | 4 | 09_110309A\110309A |
| 5 | SS33614 | Calibration Standard, 2.0 ng/mL | 5 | 09_110309A\110309A |
| | SS33613 | Calibration Standard, 5.0 ng/mL | 6 | 09_110309A\110309A |
| | Methanol Wash | Methanol Wash | 91 | 09_110309A\110309A |
| 8 | Methanol Wash | Methanol Wash | 91 | 09_110309A\110309A |
| | Control | MC4311 Deer Serum Control | 41 | 09_110309A\110309A |
| | LCS A | MC4311 Deer Serum Spike A, 10 ng/mL | 42 | 09_110309A\110309A |
| 11 | LCS B | MC4311 Deer Serum Spike B, 50 ng/mL | 43 | 09_110309A\110309A |
| 2 | L19345-2 Spk C | Deer # 7 3.5 yr male-serum Spike C, 50 ng/mL | 44 | 09_110309A\110309A |
| 13 | L19345-2 | Deer # 7 3.5 yr male-serum | 45 | 09_110309A\110309A |
| 14 | L19345-2 Dup | Deer # 7 3.5 yr male-serum Duplicate | 46 | 09_110309A\110309A |
| 5 | L19345-1 | Deer # 6 0.5 yr female-serum | 47 | 09_110309A\110309A |
| 16 | SS33615 | CCV, 1.0 ng/mL | 4 | 09_110309A\110309A |

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Protocol No: NA

MPI Study No: L19345

Instrument: AB API 4000 Biomolecular Mass Analyzer, (LC/MS/MS #9)
SCIEX Turbo Ion Spray Liquid Introduction Interface
Turbo Ion spray temperature = 450 °C

Computer: Dell OptiPlex GX 110

Software: PE Sciex Analyst 1.4

HPLC Equipment: Hewlett Packard (HP) Series 1100
HP Quat Pump HP Vacuum Degasser
HP Autosampler HP Column Oven

HPLC Column: Phenomenex Luna C8 (2) Mercury, 2cm x 4mm, 3 µm (ExyLIMS ID:
MA0052622)

Column Temperature: 35°C

Mobile Phase (A): 2 mM Ammonium Acetate in Water (ExyLIMS ID: SL0045925)

Mobile Phase (B): Methanol (ExyLIMS ID: RE0047880)

Injected Volume: 15 µL

| Time (min) | % A | % B | Flow Rate (µL/min) |
|------------|-----|-----|-----------------------|
| 0.0 | 90 | 10 | 750 |
| 0.5 | 90 | 10 | 750 |
| 2.0 | 10 | 90 | 750 |
| 5.0 | 10 | 90 | 750 |
| 5.1 | 0 | 100 | 750 |
| 6.0 | 0 | 100 | 750 |
| 6.1 | 90 | 10 | 750 |
| 10.0 | 90 | 10 | 750 |

Ions monitored:

| Analyte | Parent ion | Daughter ion(s) | Dwell (secs) |
|----------------------------|------------|-----------------|--------------|
| PFOA | 413 | 369 | 0.200 |
| PFOS | 499 | 80 | 0.200 |
| ¹³ C PFOA (m+2) | 415 | 370 | 0.200 |
| Internal Standard | | | |
| ¹³ C PFOS (m+4) | 503 | 80 | 0.200 |
| Internal Standard | | | |

Analyst: Mark Neeley *MDR 11-3-09*
MPI Research, Inc.
3058 Research Drive, State College, PA 16801
Phone: (814) 272-1039 FAX: (814) 231-1580

All Handwritten Peak ID's by: *MDR 11-6-09*
Mark Neeley

MDR 11-6-09

alyst Version: 1.4.2
rinting Time: 2:19:43 PM
rinting Date: Tuesday, November 03, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MDN 11-3-09

Acquisition Information:

quisition Method: P5195_102909.dam
reated: Thursday October 29 2009 09: 35: 12 AM
Last Modified: Friday October 30 2009 10: 56: 20 AM
Comment:
ynchronization Mode: PFCA PFCS
uto-Equilibration: LC Sync
Acquisition Duration: Off
Number Of Scans: 10min0sec
eriods In File: 732
Acquisition Module: 1
Software version: Acquisition Method
Analyst 1.4.2

Period 1:

Scans in Period: 732
relative Start Time: 0.00 msec
Experiments in Period: 1

eriod 1 Experiment 1:

Scan Type: MRM (MRM)
Polarity: Negative
can Mode: N/A
on Source: Turbo Spray
Resolution Q1: Unit
Resolution Q3: Unit
ntensity Thres.: 0.00 cps
Settling Time: 0.0000 msec
MR Pause: 5.0070 msec
CA: No
tep Size: 0.00 amu

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 13.00 | 369.00 | 200.00 | DF | -32.00 | -32.00 |
| | | | CE | -18.00 | -18.00 |
| | | | CXF | -13.20 | -13.20 |

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 415.00 | 370.00 | 200.00 | DF | -32.00 | -32.00 |
| | | | CE | -18.00 | -18.00 |
| | | | CXF | -13.20 | -13.20 |

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 499.00 | 80.00 | 200.00 | DF | -83.00 | -83.00 |
| | | | CE | -88.00 | -88.00 |
| | | | CXF | -6.00 | -6.00 |

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 503.00 | 80.00 | 200.00 | DF | -83.00 | -83.00 |
| | | | CE | -88.00 | -88.00 |

alyst Version: 1.4.2
rinting Time: 2:19:43 PM

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

rinting Date: Tuesday, November 03, 2009

MON 11-3-09

CXF -6.00 -6.00

Parameter Table (Period 1 Experiment 1):

AD: 7.00
CUR: 20.00
GS1: 50.00
S2: 40.00
S: -4500.00
TEM: 450.00
the: ON
E: -10.00

Agilent LC Pump Method Properties

Pump Model: Agilent 1100 LC Quaternary Pump
Minimum Pressure (psi): 0.0
Maximum Pressure (psi): 5801.0
Compressibility: 100.0
Dead Volume (µl): 40.0
Stroke Volume (µl): -1.0
Maximum Flow Ramp (ml/min²): 100.0
Maximum Pressure Ramp (psi/sec): 290.0

Step Table:

| Step | Total Time (min) | Flow Rate (µl/min) | A (%) | B (%) | C (%) | D (%) | TE#1 | TE#2 | TE#3 | TE#4 |
|------|------------------|--------------------|-------|-------|-------|-------|------|------|------|------|
| 0 | 0.00 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| | 0.50 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| | 2.00 | 750 | 0.0 | 0.0 | 10.0 | 90.0 | open | open | open | open |
| 3 | 5.00 | 750 | 0.0 | 0.0 | 10.0 | 90.0 | open | open | open | open |
| 4 | 5.10 | 750 | 0.0 | 0.0 | 0.0 | 100.0 | open | open | open | open |
| | 6.00 | 750 | 0.0 | 0.0 | 0.0 | 100.0 | open | open | open | open |
| | 6.10 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| 7 | 10.00 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |

Primary Flow Rate (µl/min): 200.0

Flow Sensor Calibration Table Index: 0

Agilent Column Oven Properties

Left Temperature (°C): 35.00
Right Temperature (°C): 35.00
Temperature Tolerance +/- (°C): 1.00
Start Acquisition Tolerance +/- (°C): 0.50

Time Table (Not Used)

Column Switching Valve Installed

Position for first sample in the batch: Left
same position for all samples in the batch

Agilent Autosampler Properties

Autosampler Model: Agilent 1100 Wellplate Autosampler
Syringe Size (µl): 100
Injection Volume (µl): 15.00
Draw Speed (µl/min): 200.0
Inject Speed (µl/min): 200.0
Needle Level (mm): 0.00
Temperature Control Enabled
Setpoint (4 - 40 C): 4
Rinse Location: Flush Port
Rinse Time (1 - 999 sec): 10

LC Delay Volume Reduction

Not Used

Research, Inc.

alyst Version: 1.4.2
rinting Time: 2:19:43 PM
rinting Date: Tuesday, November 03, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MDL 11-3-uf

Equilibration Time (sec): 2
Enable Vial/Well Bottom Sensing No
se Custom Injector Program No

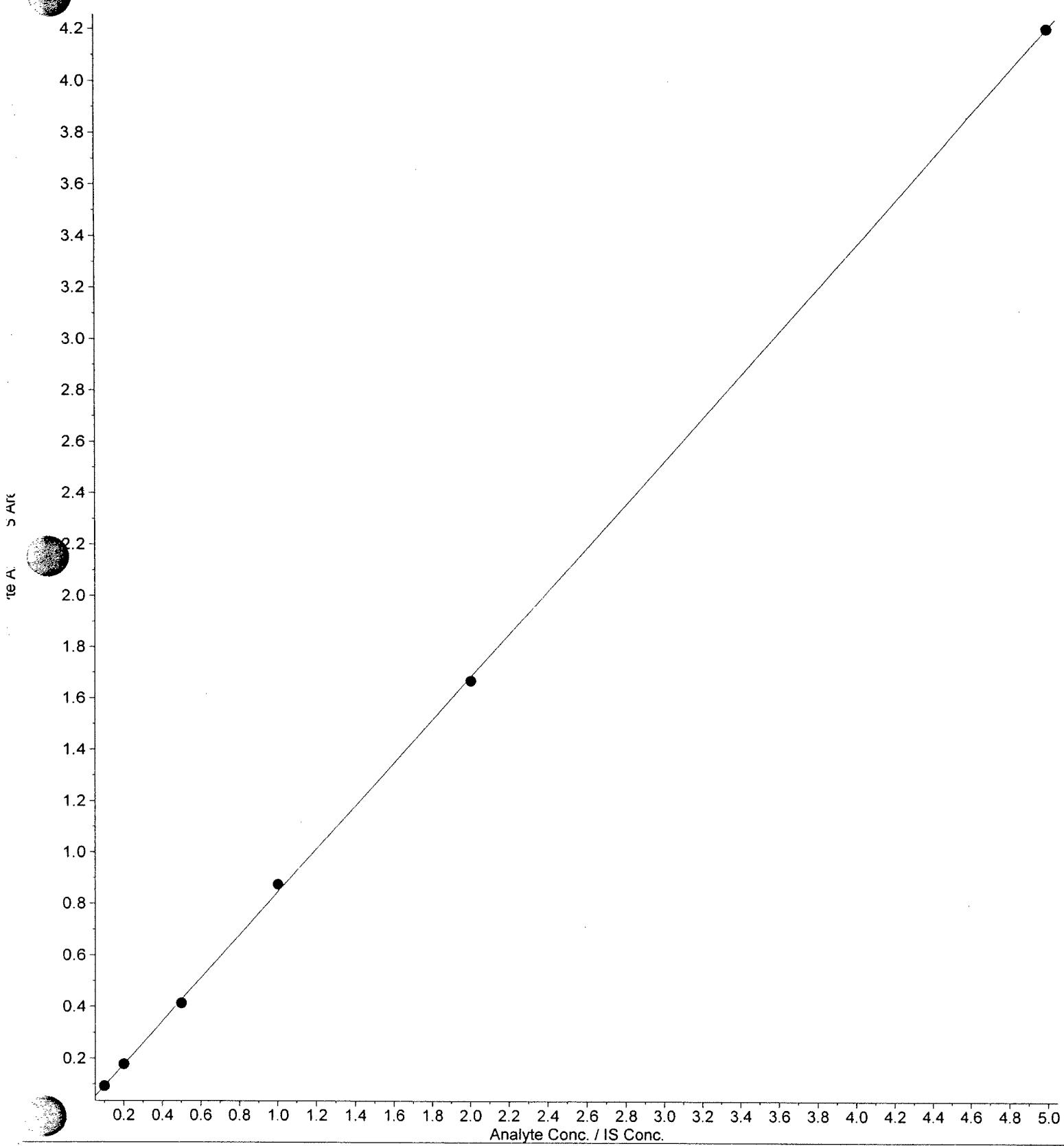
lyst Version: 1.4.2
rinting Time: 8:54:46 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MS/MS 11-6-09

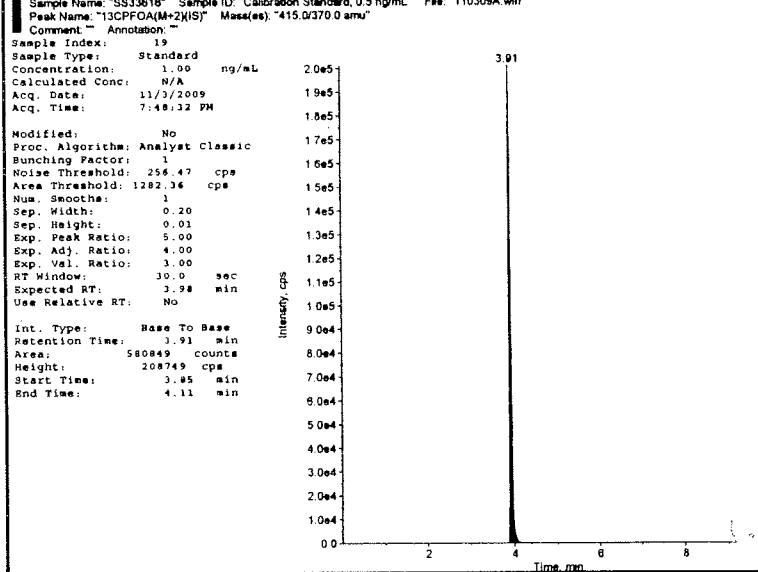
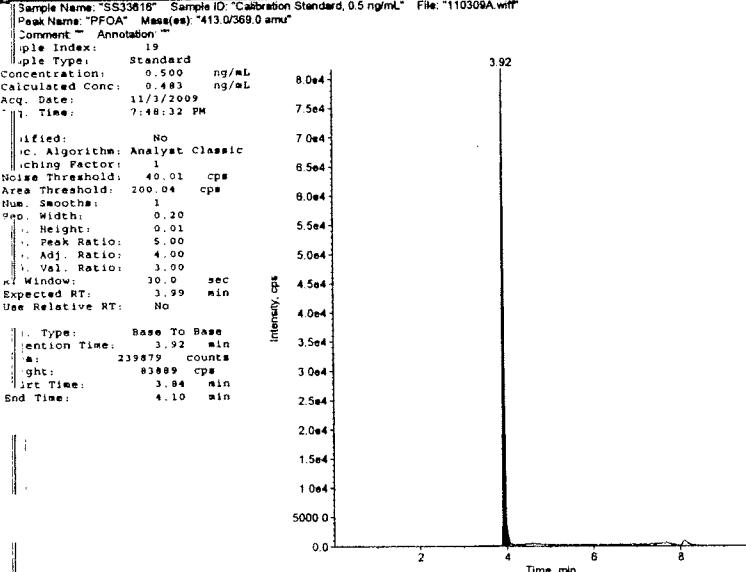
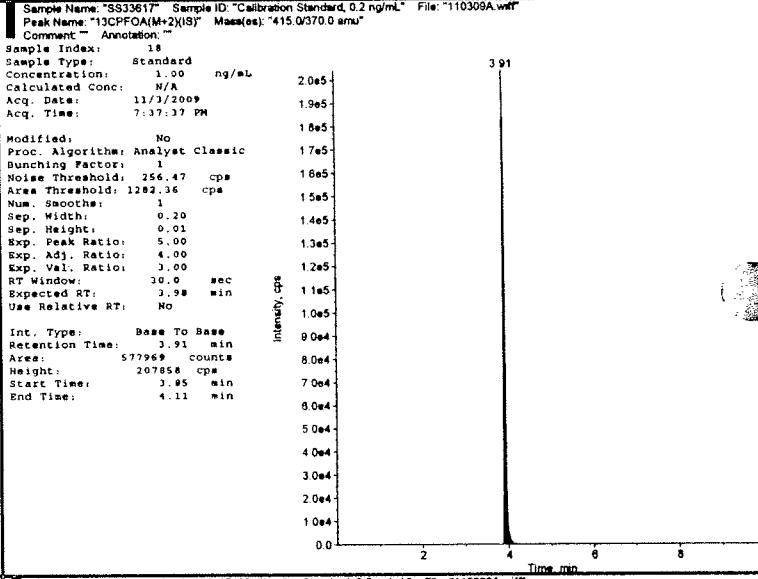
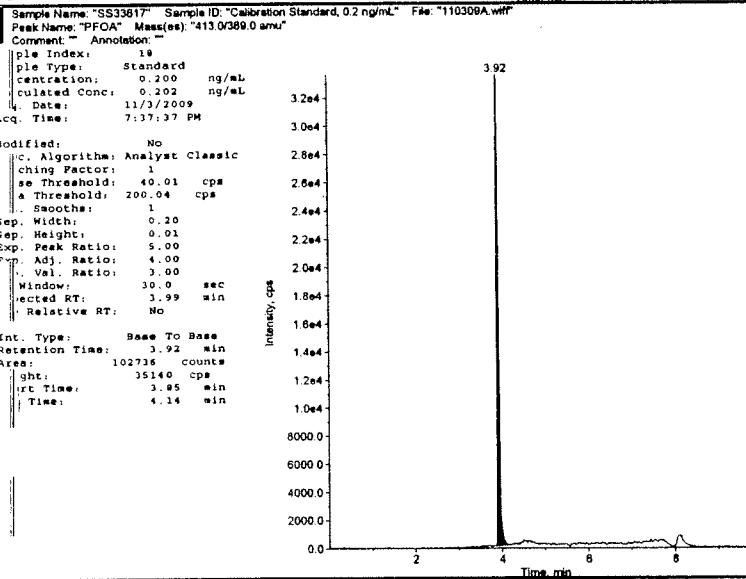
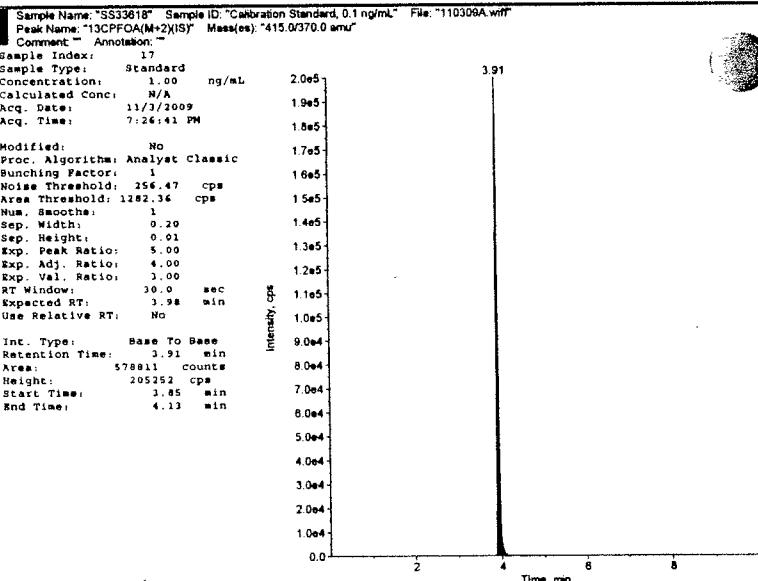
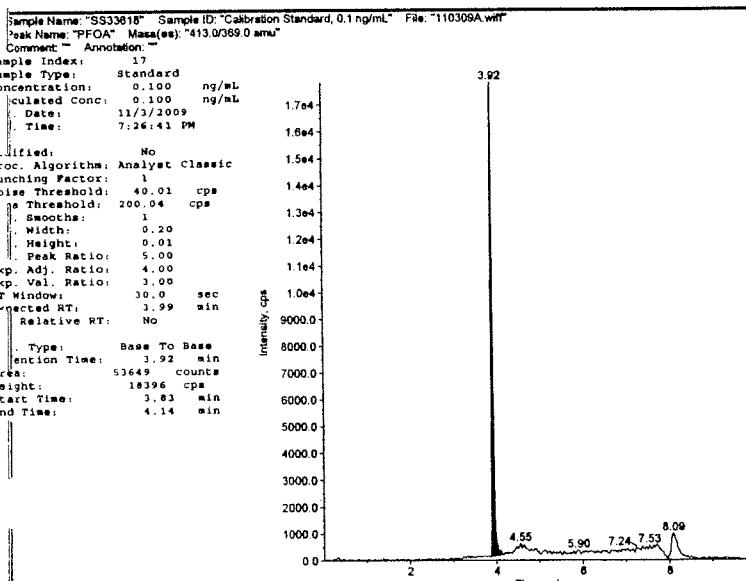
09_110309A.rdb (PFOA): "Linear" Regression ("1 / x" weighting): $y = 0.838x + 0.00876$ ($r = 0.9999$)



lyst Version: 1.4.2
rinting Time: 8:55:27 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9



M Research, Inc.

Initials MDJ

Date 11-6-09

Sample 17

Index 32

ilyst Version: 1.4.2

rinting Time: 8:55:27 AM

rinting Date: Friday, November 06, 2009

MPI Study: L19345

MPI Set No.: 110309A

Operator: Mark Neeley

Instrument No.: LC/MS/MS #9

Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0369.0 amu"

Comment: " Annotation: "

Sample Index: 23

Sample Type: Solvent

Conc.: 0.00 ng/mL

Calcd. Conc.: N/A

Acq. Date: 11/3/2009

Acq. Time: 8:32:16 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Relative RT: No

Type: Base To Base

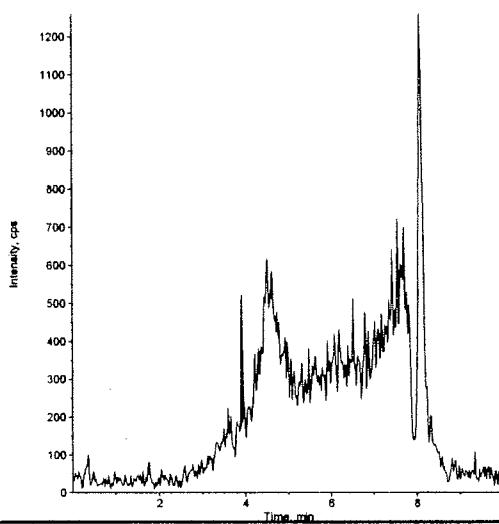
Retention Time: 3.99 min

Area: 1023 counts

Height: 348 cps

Start Time: 3.87 min

End Time: 3.99 min



Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)(S)" Mass(es): "415.0370.0 amu"

Comment: " Annotation: "

Sample Index: 23

Sample Type: Solvent

Conc.: 1.00 ng/mL

Calcd. Conc.: N/A

Acq. Date: 11/3/2009

Acq. Time: 8:32:16 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Relative RT: No

Type: Base To Base

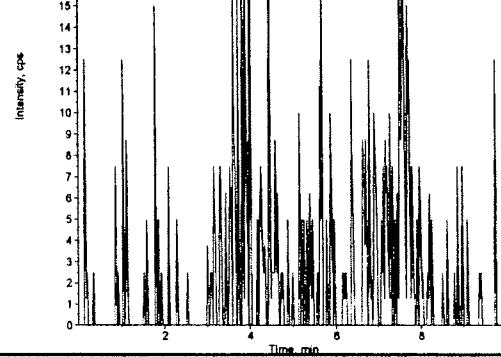
Retention Time: 3.99 min

Area: 1023 counts

Height: 348 cps

Start Time: 3.87 min

End Time: 3.99 min



Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0369.0 amu"

Comment: " Annotation: "

Sample Index: 24

Sample Type: Solvent

Concentration: 0.00 ng/mL

Calcd. Conc.: N/A

Acq. Date: 11/3/2009

Acq. Time: 8:43:12 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Relative RT: No

Type: Base To Base

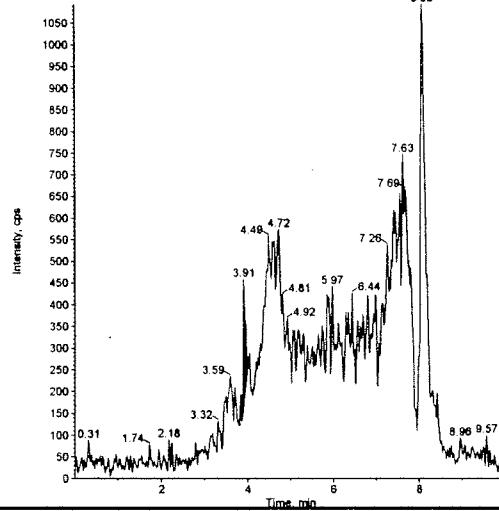
Retention Time: 3.99 min

Area: 1068 counts

Height: 290 cps

Start Time: 3.87 min

End Time: 3.99 min



Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)(S)" Mass(es): "415.0370.0 amu"

Comment: " Annotation: "

Sample Index: 24

Sample Type: Solvent

Concentration: 0.00 ng/mL

Calcd. Conc.: N/A

Acq. Date: 11/3/2009

Acq. Time: 8:43:12 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Relative RT: No

Type: Base To Base

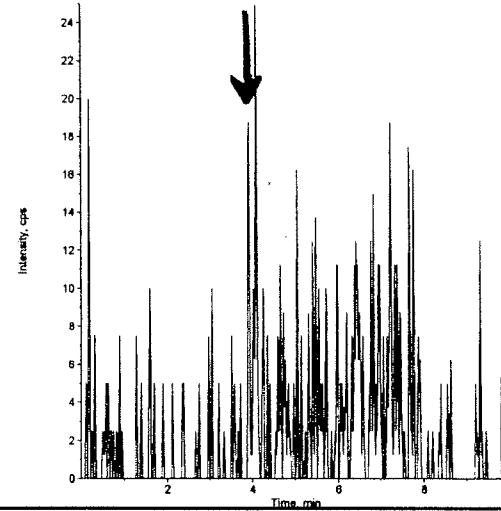
Retention Time: 3.99 min

Area: 1068 counts

Height: 290 cps

Start Time: 3.87 min

End Time: 3.99 min



Sample Name: "Control" Sample ID: "MC4311 Deer Serum Control" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0369.0 amu"

Comment: " Annotation: "

Sample Index: 25

Sample Type: Unknown

Concentration: N/A

Calcd. Conc: < 0

Acq. Date: 11/3/2009

Acq. Time: 8:54:07 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Relative RT: No

Type: Base To Base

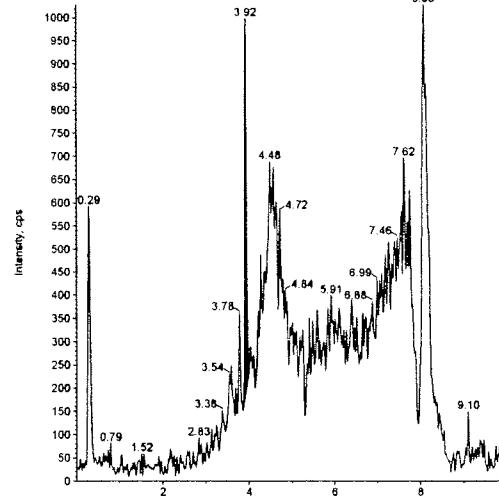
Retention Time: 3.92 min

Area: 2392 counts

Height: 833 cps

Start Time: 3.85 min

End Time: 3.99 min



Sample Name: "Control" Sample ID: "MC4311 Deer Serum Control" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)(S)" Mass(es): "415.0370.0 amu"

Comment: " Annotation: "

Sample Index: 25

Sample Type: Unknown

Concentration: 0.500 ng/mL

Calcd. Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 8:54:07 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 cps

Area Threshold: 1282.36 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Relative RT: No

Type: Base To Base

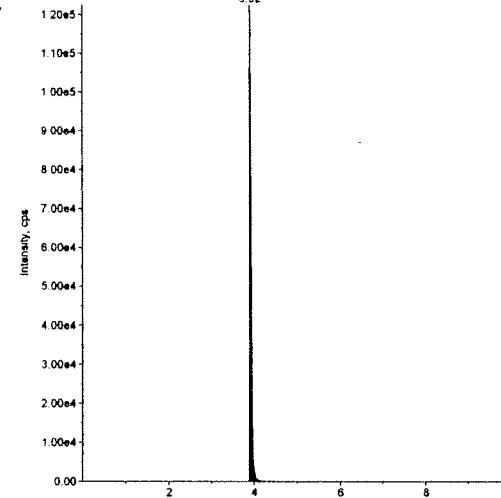
Retention Time: 3.92 min

Area: 347101 counts

Height: 129136 cps

Start Time: 3.87 min

End Time: 4.09 min



alyst Version: 1.4.2
rinting Time: 8:55:27 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Sample Name: "LCS A" Sample ID: "MC4311 Deer Serum Spike A, 10 ng/mL" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 26

Sample Type: QC

Concentration: 10.0 ng/mL

Calculated Conc: 10.1 ng/mL

i. Date: 11/3/2009

Acq. Time: 9:05:03 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

x. Smooths: 1

sep. Width: 0.20

sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 154309 counts

Height: 56763 cps

Start Time: 3.81 min

End Time: 4.11 min

Intensity, cps

5000.0

0.0

Time, min

Sample Name: "LCS B" Sample ID: "MC4311 Deer Serum Spike B, 50 ng/mL" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 27

Sample Type: QC

Concentration: 50.0 ng/mL

Calculated Conc: 47.7 ng/mL

i. Date: 11/3/2009

Acq. Time: 9:16:01 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

x. Smooths: 1

sep. Width: 0.20

sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.92 min

Area: 751597 counts

Height: 270613 cps

Start Time: 3.83 min

End Time: 4.18 min

Intensity, cps

8.0e4

6.0e4

4.0e4

2.0e4

0.0

Time, min

Sample Name: "L19345-2 Spk C" Sample ID: "Deer # 7.5 yr male-serum Spike C, 50 ng/mL" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 28

Sample Type: QC

Concentration: 50.0 ng/mL

Calculated Conc: 51.0 ng/mL

i. Date: 11/3/2009

Acq. Time: 9:26:57 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 cps

Area Threshold: 200.04 cps

x. Smooths: 1

sep. Width: 0.20

sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 822293 counts

Height: 305382 cps

Start Time: 3.85 min

End Time: 4.16 min

Intensity, cps

8.0e4

6.0e4

4.0e4

2.0e4

0.0

Time, min

Sample Name: "LCS A" Sample ID: "MC4311 Deer Serum Spike A, 10 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)IS" Mass(es): "415.0/370.0 amu"

Comment: " Annotation: "

Sample Index: 26

Sample Type: QC

Concentration: 0.500 ng/mL

Calculated Conc: N/A

i. Date: 11/3/2009

Acq. Time: 9:05:03 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 cps

Area Threshold: 1282.36 cps

x. Smooths: 1

sep. Width: 0.20

sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 355719 counts

Height: 132129 cps

Start Time: 3.85 min

End Time: 4.09 min

Intensity, cps

6.0e4

5.0e4

4.0e4

3.0e4

2.0e4

1.0e4

0.0

Time, min

Sample Name: "LCS B" Sample ID: "MC4311 Deer Serum Spike B, 50 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)IS" Mass(es): "415.0/370.0 amu"

Comment: " Annotation: "

Sample Index: 27

Sample Type: QC

Concentration: 0.500 ng/mL

Calculated Conc: N/A

i. Date: 11/3/2009

Acq. Time: 9:16:01 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 cps

Area Threshold: 1282.36 cps

x. Smooths: 1

sep. Width: 0.20

sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 355719 counts

Height: 132129 cps

Start Time: 3.85 min

End Time: 4.09 min

Intensity, cps

6.0e4

5.0e4

4.0e4

3.0e4

2.0e4

1.0e4

0.0

Time, min

Sample Name: "L19345-2 Spk C" Sample ID: "Deer # 7.5 yr male-serum Spike C, 50 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)IS" Mass(es): "415.0/370.0 amu"

Comment: " Annotation: "

Sample Index: 28

Sample Type: QC

Concentration: 0.500 ng/mL

Calculated Conc: N/A

i. Date: 11/3/2009

Acq. Time: 9:26:57 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 cps

Area Threshold: 1282.36 cps

x. Smooths: 1

sep. Width: 0.20

sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 383265 counts

Height: 141758 cps

Start Time: 3.85 min

End Time: 4.07 min

Intensity, cps

6.0e4

5.0e4

4.0e4

3.0e4

2.0e4

1.0e4

0.0

Time, min

alyst Version: 1.4.2

rinting Time: 8:55:27 AM

rinting Date: Friday, November 06, 2009

MPI Study: L19345

MPI Set No.: 110309A

Operator: Mark Neeley

Instrument No.: LC/MS/MS #9

Sample Name: "L19345-2" Sample ID: "Deer #7 3.5 yr male-serum" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 29

Sample Type: Unknown

Concentration: N/A

Calculated Conc: 0.991 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:37:54 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 CPS

Area Threshold: 200.04 CPS

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 18517 counts

Height: 7105 CPS

Start Time: 3.84 min

End Time: 3.99 min

Intensity, CPS

3.91

Sample Name: "L19345-2 Dup" Sample ID: "Deer #7 3.5 yr male-serum Duplicate" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 30

Sample Type: Unknown

Concentration: N/A

Calculated Conc: 0.983 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:48:52 PM

Modified: Yes

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 80.49 CPS

Area Threshold: 402.43 CPS

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.92 min

Relative RT: No

Int. Type: Base To Base

Retention Time: 3.92 min

Area: 20592 counts

Height: 7630 CPS

Start Time: 3.85 min

End Time: 4.00 min

Intensity, CPS

3.92

Sample Name: "L19345-1" Sample ID: "Deer #8 0.5 yr female-serum" File: "110309A.wif"

Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"

Comment: " Annotation: "

Sample Index: 31

Sample Type: Unknown

Concentration: N/A

Calculated Conc: 0.287 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:59:48 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 40.01 CPS

Area Threshold: 200.04 CPS

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.99 min

Relative RT: No

Int. Type: Base To Base

Retention Time: 3.92 min

Area: 7527 counts

Height: 2707 CPS

Start Time: 3.85 min

End Time: 4.00 min

Intensity, CPS

3.92

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Sample Name: "L19345-2" Sample ID: "Deer #7 3.5 yr male-serum" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)(S)" Mass(es): "415.0/370.0 amu"

Comment: " Annotation: "

Sample Index: 29

Sample Type: Unknown

Concentration: N/A

Calculated Conc: 0.991 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:37:54 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 CPS

Area Threshold: 1282.36 CPS

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 36833 counts

Height: 135272 CPS

Start Time: 3.85 min

End Time: 4.10 min

Intensity, CPS

3.91

Sample Name: "L19345-2 Dup" Sample ID: "Deer #7 3.5 yr male-serum Duplicate" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)(S)" Mass(es): "415.0/370.0 amu"

Comment: " Annotation: "

Sample Index: 30

Sample Type: Unknown

Concentration: 0.500 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 9:48:52 PM

Modified: Yes

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 CPS

Area Threshold: 1282.36 CPS

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 412407 counts

Height: 154890 CPS

Start Time: 3.85 min

End Time: 4.10 min

Intensity, CPS

3.91

Sample Name: "L19345-1" Sample ID: "Deer #8 0.5 yr female-serum" File: "110309A.wif"

Peak Name: "13CPFOA(M+2)(S)" Mass(es): "415.0/370.0 amu"

Comment: " Annotation: "

Sample Index: 31

Sample Type: Unknown

Concentration: 0.500 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 9:59:48 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 256.47 CPS

Area Threshold: 1282.36 CPS

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 3.98 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.91 min

Area: 162334 counts

Height: 132313 CPS

Start Time: 3.85 min

End Time: 4.10 min

Intensity, CPS

3.91

lyst Version: 1.4.2
rinting Time: 8:55:27 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Sample Name: "SS33815" Sample ID: "CCV, 1.0 ng/mL" File: "110309A.wif"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu"
Comment: " Annotation: "
Sample Index: 32
Sample Type: QC
Concentration: 1.00 ng/mL
Calculated Conc: 1.02 ng/mL
Acq. Date: 11/3/2009
Acq. Time: 10:10:46 PM
Labeled: No
Proc. Algorithm: Analyst Classic 1.4e5
Bunching Factor: 1
Noise Threshold: 40.01 cps 1.3e5
Area Threshold: 200.04 cps
Smooths: 1 1.2e5
Width: 0.20
Height: 0.01 1.1e5
Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00 1.0e5
RT Window: 30.0 sec
Expected RT: 3.99 min 9.0e4
Relative RT: No 8.0e4
...
Type: Base To Base
Retention Time: 3.91 min 7.0e4
...
Height: 479482 counts
Start Time: 3.83 min 6.0e4
End Time: 4.16 min 5.0e4
4.0e4
3.0e4
2.0e4
1.0e4
0.0

3.81

Sample Name: "SS33815" Sample ID: "CCV, 1.0 ng/mL" File: "110309A.wif"
Peak Name: "13CPFOA(M+2)IS" Mass(es): "415.0/370.0 amu"
Comment: " Annotation: "
Sample Index: 32
Sample Type: QC
Concentration: 1.00 ng/mL
Calculated Conc: N/A 1.9e5
Acq. Date: 11/3/2009
Acq. Time: 10:10:46 PM 1.8e5
Modified: No 1.7e5
Proc. Algorithm: Analyst Classic 1.8e5
Bunching Factor: 1
Noise Threshold: 256.47 cps 1.5e5
Area Threshold: 1242.36 cps
Num. Smooths: 1 1.4e5
Sep. Type: 0.20
Exp. Height: 0.01 1.3e5
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00 1.2e5
Exp. Val. Ratio: 3.00
RT Window: 30.0 sec 8 1.1e5
Expected RT: 3.98 min 1.0e5
Use Relative RT: No 9.0e4
...
Int. Type: Base To Base 8.0e4
Retention Time: 3.91 min 7.0e4
Area: 556775 counts 6.0e4
Height: 197412 cps 5.0e4
Start Time: 3.85 min 4.0e4
End Time: 4.11 min 3.0e4
2.0e4
1.0e4
0.0

3.81

Intensity, cps

Intensity, cps

Time, min

Time, min

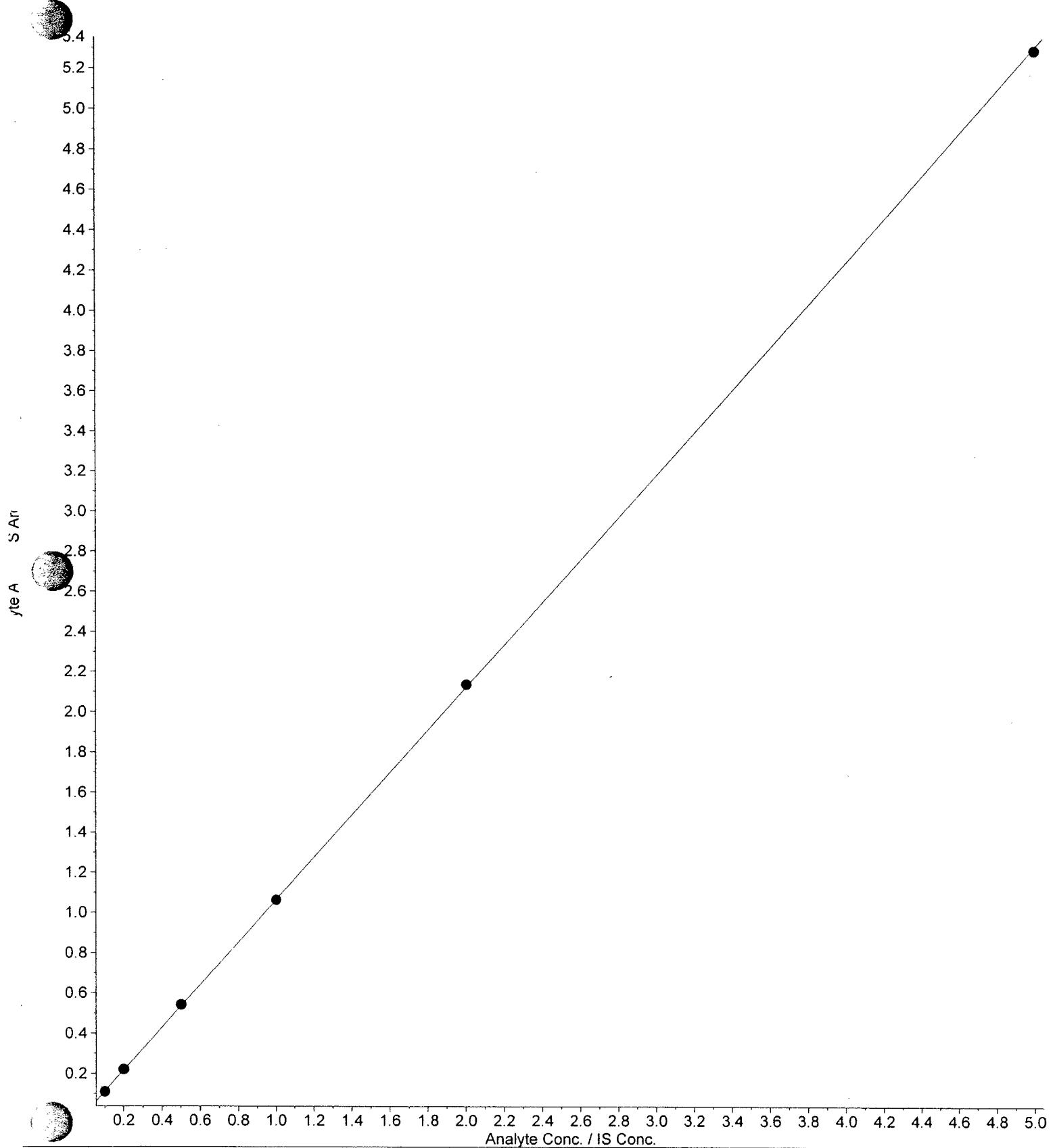
lyst Version: 1.4.2
rinting Time: 8:54:59 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MDR 11-6-09

09_110309A.rdb (PFOS): "Linear" Regression ("1 / x" weighting): $y = 1.06x + 0.00618$ ($r = 1.0000$)



lyst Version: 1.4.2
rinting Time: 8:55:46 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Sample Name: "SS33618" Sample ID: "Calibration Standard, 0.1 ng/mL" File: "110309A.wif"

Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comment: " Annotation: "

Sample Index: 17

Sample Type: Standard

Concentration: 0.100 ng/mL

Calculated Conc: 0.0979 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 7:26:41 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

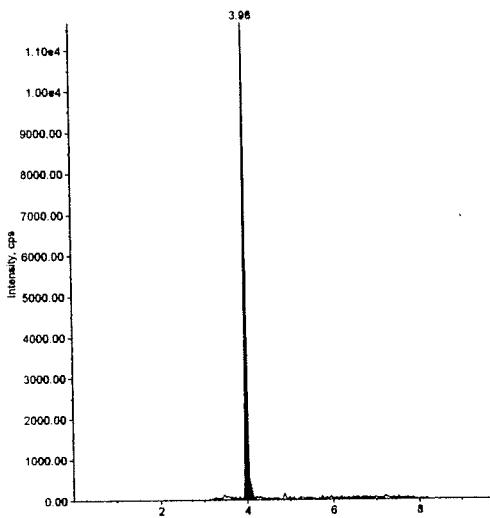
Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 10.0 sec

Expected RT: 4.06 min

Relative RT: No



Sample Name: "SS33618" Sample ID: "Calibration Standard, 0.1 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOS(M+4)(S)" Mass(es): "503.0/60.0 amu"

Comment: " Annotation: "

Sample Index: 17

Sample Type: Standard

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 7:26:41 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

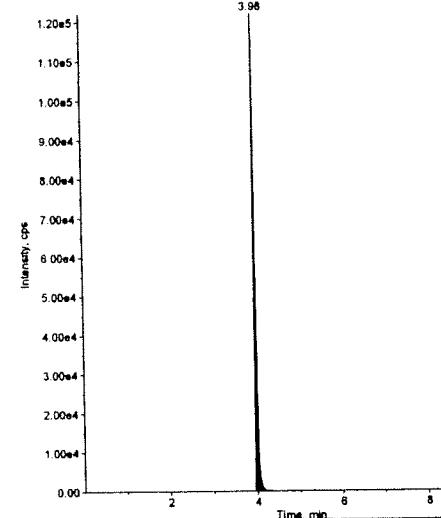
Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 10.0 sec

Expected RT: 4.06 min

Use Relative RT: No



Sample Name: "SS33617" Sample ID: "Calibration Standard, 0.2 ng/mL" File: "110309A.wif"

Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comment: " Annotation: "

Sample Index: 18

Sample Type: Standard

Concentration: 0.200 ng/mL

Calculated Conc: 0.202 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 7:37:37 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Int. Type: Base To Base

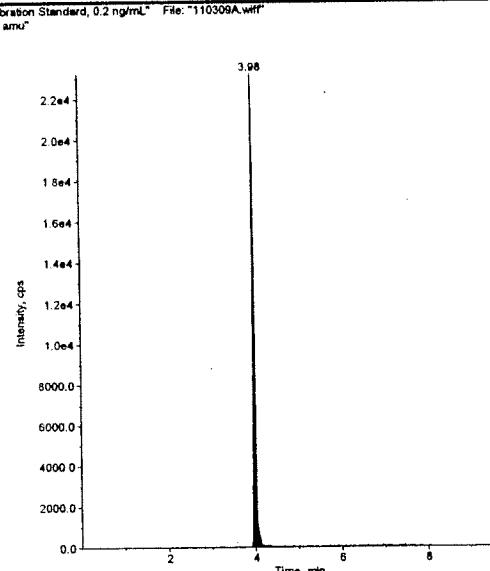
Retention Time: 3.98 min

Area: 75206 counts

Height: 13699 CPS

Start Time: 3.89 min

End Time: 4.17 min



Sample Name: "SS33617" Sample ID: "Calibration Standard, 0.2 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOS(M+4)(S)" Mass(es): "503.0/60.0 amu"

Comment: " Annotation: "

Sample Index: 19

Sample Type: Standard

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 7:37:37 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

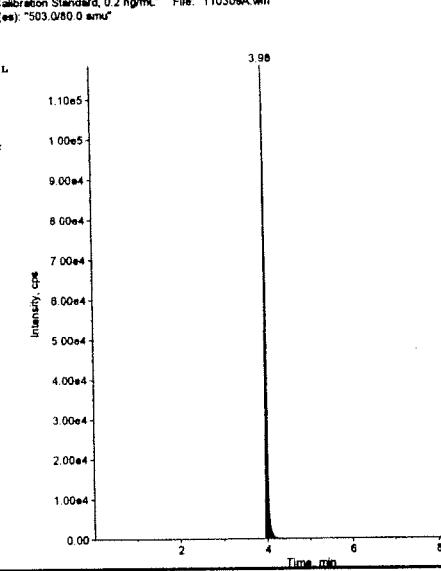
Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No



Sample Name: "SS33616" Sample ID: "Calibration Standard, 0.5 ng/mL" File: "110309A.wif"

Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comment: " Annotation: "

Sample Index: 19

Sample Type: Standard

Concentration: 0.500 ng/mL

Calculated Conc: 0.505 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 7:48:32 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

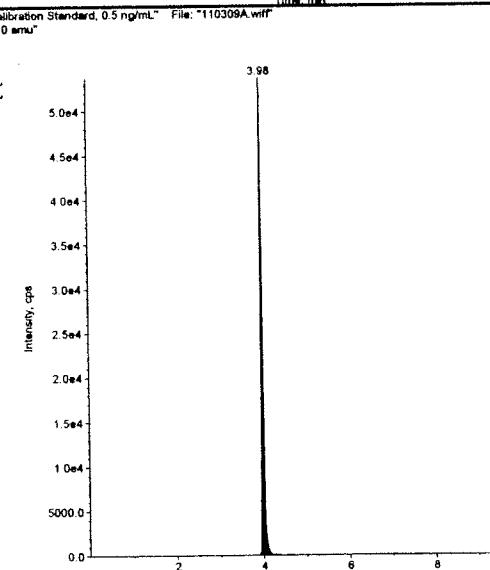
Retention Time: 3.98 min

Area: 172951 counts

Height: 55742 CPS

Start Time: 3.88 min

End Time: 4.20 min



Sample Name: "SS33616" Sample ID: "Calibration Standard, 0.5 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOS(M+4)(S)" Mass(es): "503.0/60.0 amu"

Comment: " Annotation: "

Sample Index: 19

Sample Type: Standard

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 7:48:32 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

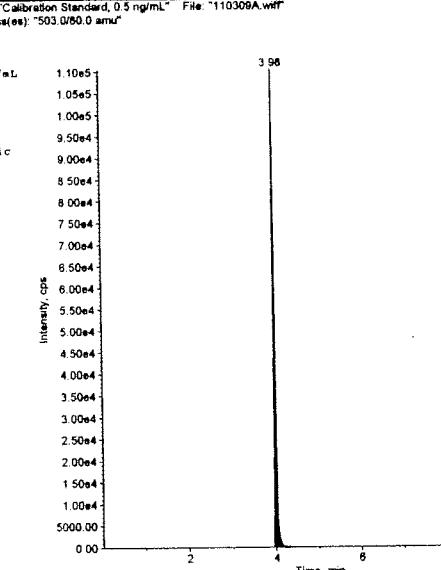
Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No



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Page 1 of 6

Initials MMD

Date 11-6-07

Sample

Index 17

To 32

lyst Version: 1.4.2
rinting Time: 8:55:46 AM

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

rinting Date: Friday, November 06, 2009

Sample Name: "SS33815" Sample ID: "Calibration Standard, 1.0 ng/mL" File: "110309A.wiff"
Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 20
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: 0.998 ng/mL
Acq. Date: 11/3/2009
Acq. Time: 7:59:27 PM
ified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 15.73 cps
Area Threshold: 78.65 cps
Smooths: 1
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00
RT Window: 30.0 sec
Expected RT: 4.06 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 3.98 min
Area: 35548 counts
Height: 111208 cps
Start Time: 3.85 min
End Time: 4.16 min
3.00e4
2.50e4
2.00e4
1.50e4
1.00e4
5000.00
0.00
Intensity, cps
Time, min

Sample Name: "SS33815" Sample ID: "Calibration Standard, 1.0 ng/mL" File: "110309A.wiff"
Peak Name: "13CPFOS(M+4)IS" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 20
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/3/2009
Acq. Time: 7:59:27 PM
ified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 140.21 cps
Area Threshold: 701.07 cps
Smooths: 1
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00
RT Window: 30.0 sec
Expected RT: 4.06 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 3.98 min
Area: 324958 counts
Height: 118647 cps
Start Time: 3.92 min
End Time: 4.18 min
3.00e4
2.00e4
1.00e4
0.00
Intensity, cps
Time, min

Sample Name: "SS33814" Sample ID: "Calibration Standard, 2.0 ng/mL" File: "110309A.wiff"
Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 21
Sample Type: Standard
Concentration: 2.00 ng/mL
Calculated Conc: 2.01 ng/mL
Acq. Date: 11/3/2009
Acq. Time: 8:10:24 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 15.73 cps
Area Threshold: 78.65 cps
Smooths: 1
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00
Window: 30.0 sec
Expected RT: 4.06 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 3.98 min
Area: 730486 counts
Height: 225121 cps
Start Time: 3.88 min
End Time: 4.28 min
1.00e4
8.00e4
6.00e4
5.00e4
4.00e4
3.00e4
2.00e4
1.00e4
0.00
Intensity, cps
Time, min

Sample Name: "SS33814" Sample ID: "Calibration Standard, 2.0 ng/mL" File: "110309A.wiff"
Peak Name: "13CPFOS(M+4)IS" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 21
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/3/2009
Acq. Time: 8:10:24 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 140.21 cps
Area Threshold: 701.07 cps
Smooths: 1
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00
RT Window: 30.0 sec
Expected RT: 4.06 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 3.98 min
Area: 342257 counts
Height: 122368 cps
Start Time: 3.92 min
End Time: 4.20 min
3.00e4
2.00e4
1.00e4
0.00
Intensity, cps
Time, min

Sample Name: "SS33813" Sample ID: "Calibration Standard, 5.0 ng/mL" File: "110309A.wiff"
Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 22
Sample Type: Standard
Concentration: 5.00 ng/mL
Calculated Conc: 4.99 ng/mL
Acq. Date: 11/3/2009
Acq. Time: 8:21:20 PM
ified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 15.73 cps
Area Threshold: 78.65 cps
Smooths: 1
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00
Window: 30.0 sec
Expected RT: 4.06 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 3.98 min
Area: 1742971 counts
Height: 546364 cps
Start Time: 3.88 min
End Time: 4.41 min
1.50e5
1.00e5
5.00e4
0.00
Intensity, cps
Time, min

Sample Name: "SS33813" Sample ID: "Calibration Standard, 5.0 ng/mL" File: "110309A.wiff"
Peak Name: "13CPFOS(M+4)IS" Mass(es): "503.0/80.0 amu"

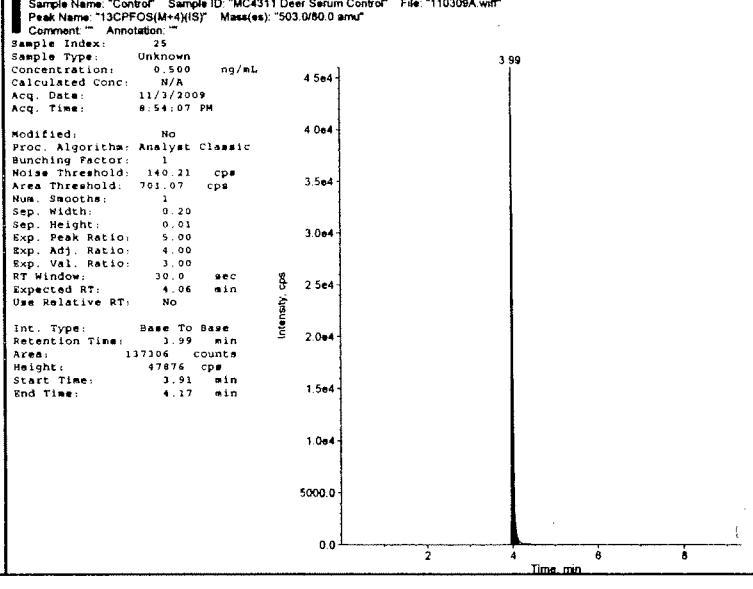
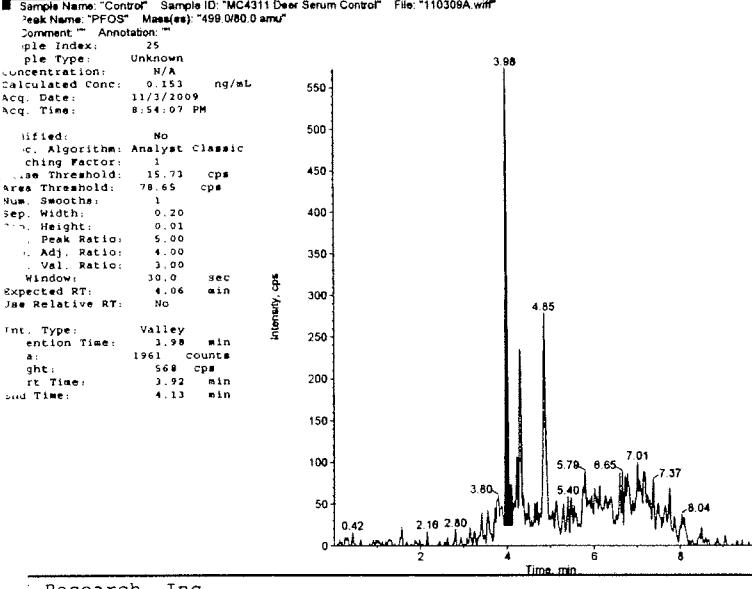
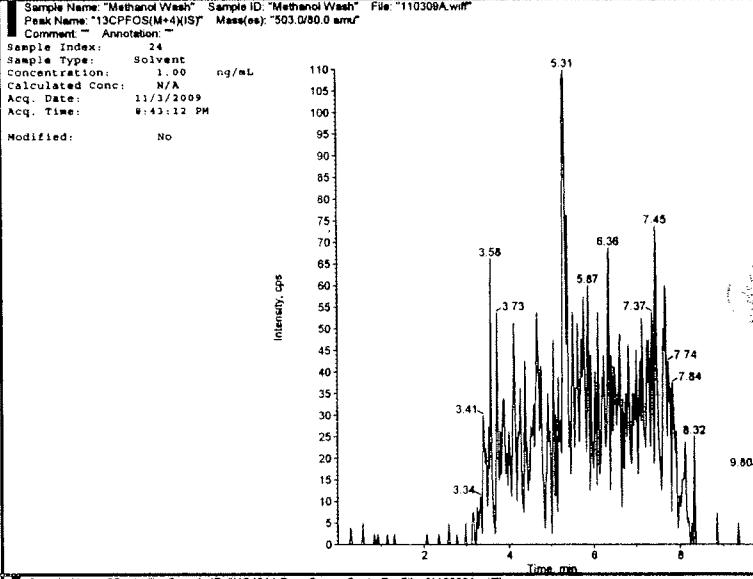
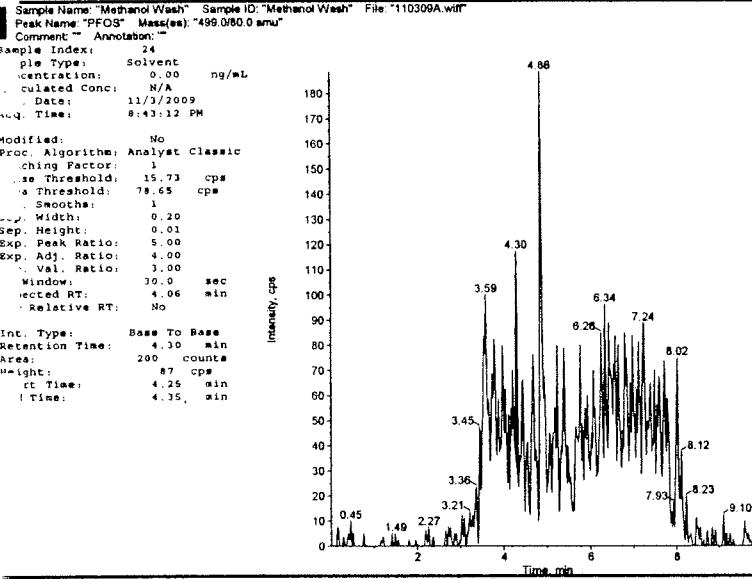
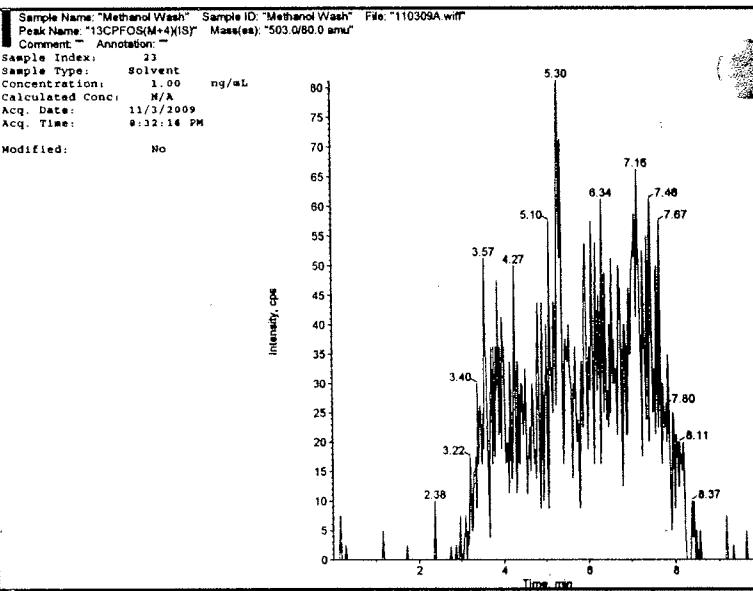
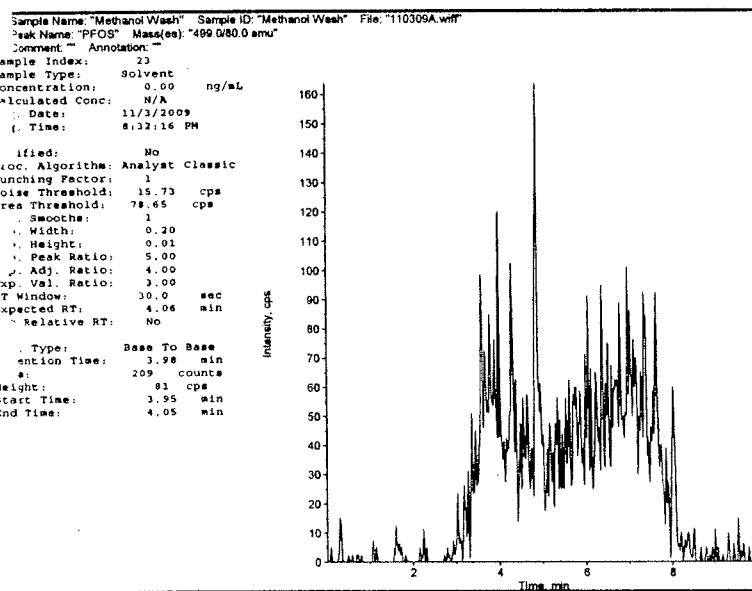
Comment: " Annotation: "

Sample Index: 22
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/3/2009
Acq. Time: 8:21:20 PM
Modified: No
Proc. Algorithm: Analyst Classic
Bunching Factor: 1
Noise Threshold: 140.21 cps
Area Threshold: 701.07 cps
Smooths: 1
Sep. Width: 0.20
Sep. Height: 0.01
Exp. Peak Ratio: 5.00
Exp. Adj. Ratio: 4.00
Exp. Val. Ratio: 3.00
RT Window: 30.0 sec
Expected RT: 4.06 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 3.98 min
Area: 329962 counts
Height: 119223 cps
Start Time: 3.92 min
End Time: 4.18 min
3.00e4
2.00e4
1.00e4
0.00
Intensity, cps
Time, min

alyst Version: 1.4.2
rinting Time: 8:55:47 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9



lyst Version: 1.4.2
rinting Time: 8:55:47 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Sample Name: "LCS A" Sample ID: "MC4311 Deer Serum Spike A, 10 ng/mL" File: "110309A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 26

Sample Type: QC

Concentration: 10.0 ng/mL

Calculated Conc: 10.0 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:05:03 PM

ified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Type: Valley

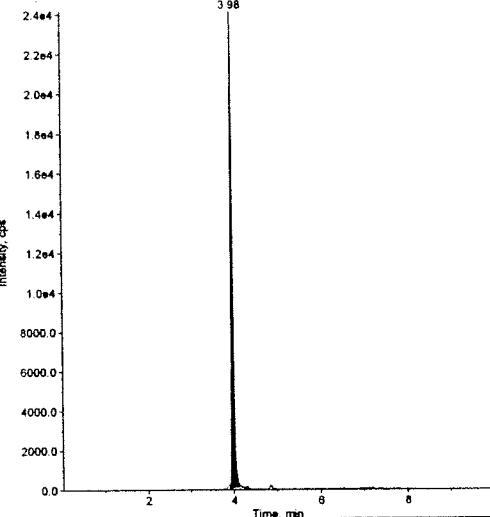
Retention Time: 3.98 min

Area: 76698 counts

Height: 24304 cps

Start Time: 3.90 min

End Time: 4.13 min



Sample Name: "LCS A" Sample ID: "MC4311 Deer Serum Spike A, 10 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOS(M+X)IS" Mass(es): "503.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 26

Sample Type: QC

Concentration: 0.500 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 9:05:03 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

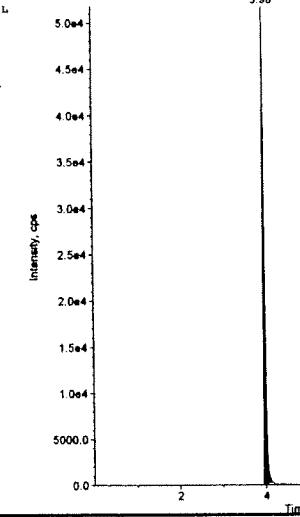
Retention Time: 3.98 min

Area: 143222 counts

Height: 52574 cps

Start Time: 3.92 min

End Time: 4.14 min



Sample Name: "LCS B" Sample ID: "MC4311 Deer Serum Spike B, 50 ng/mL" File: "110309A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 27

Sample Type: QC

Concentration: 50.0 ng/mL

Calculated Conc: 49.0 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:16:01 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Type: Exponential Skim

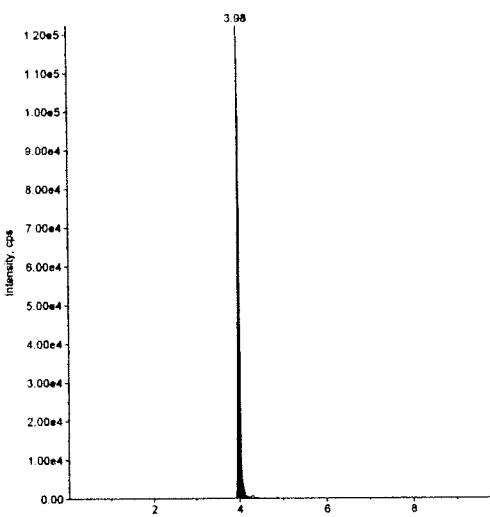
Retention Time: 3.98 min

Area: 404559 counts

Height: 124526 cps

Start Time: 3.93 min

End Time: 4.14 min



Sample Name: "LCS B" Sample ID: "MC4311 Deer Serum Spike B, 50 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOS(M+X)IS" Mass(es): "503.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 27

Sample Type: QC

Concentration: 0.500 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 9:16:01 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

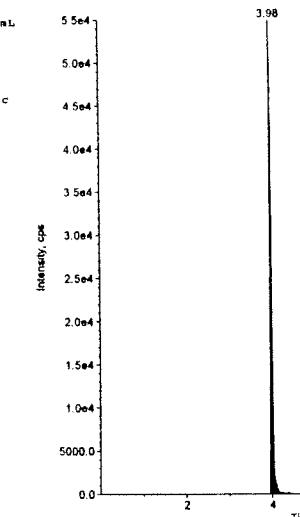
Retention Time: 3.98 min

Area: 154891 counts

Height: 56246 cps

Start Time: 3.92 min

End Time: 4.16 min



Sample Name: "L19345-2 Spk C" Sample ID: "Deer #7 3.5 yr male-serum Spike C, 50 ng/mL" File: "110309A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 28

Sample Type: QC

Concentration: 50.0 ng/mL

Calculated Conc: 88.2 ng/mL

Acq. Date: 11/3/2009

Acq. Time: 9:26:57 PM

ified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Type: Valley

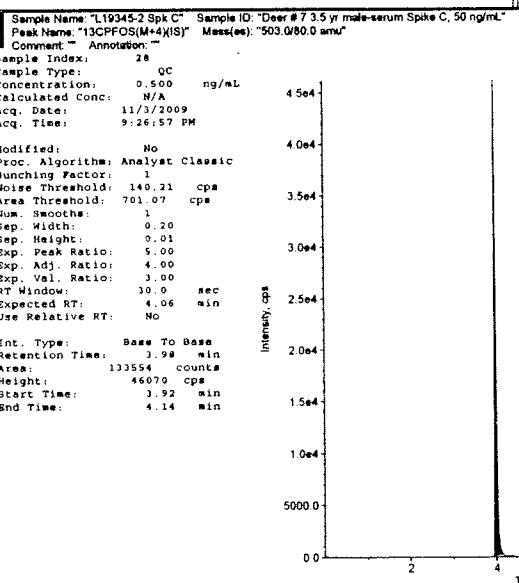
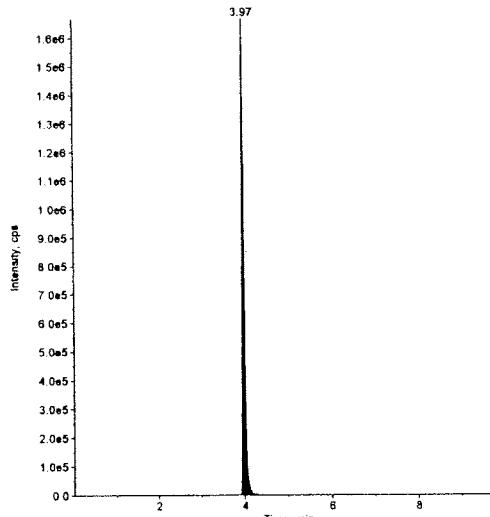
Retention Time: 3.97 min

Area: 6210547 counts

Height: 1678561 cps

Start Time: 3.81 min

End Time: 4.61 min

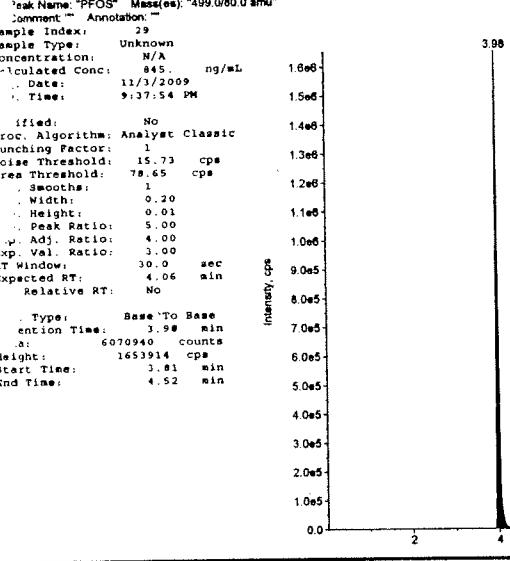


lyst Version: 1.4.2
rinting Time: 8:55:47 AM
rinting Date: Friday, November 06, 2009

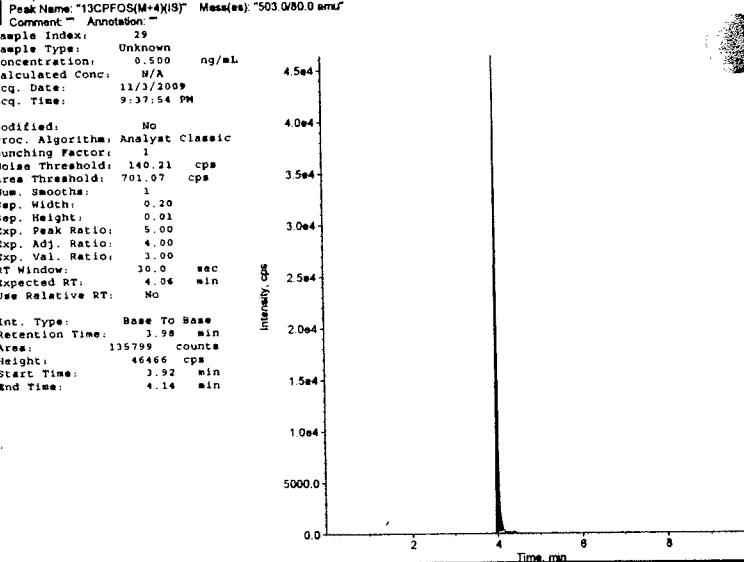
MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

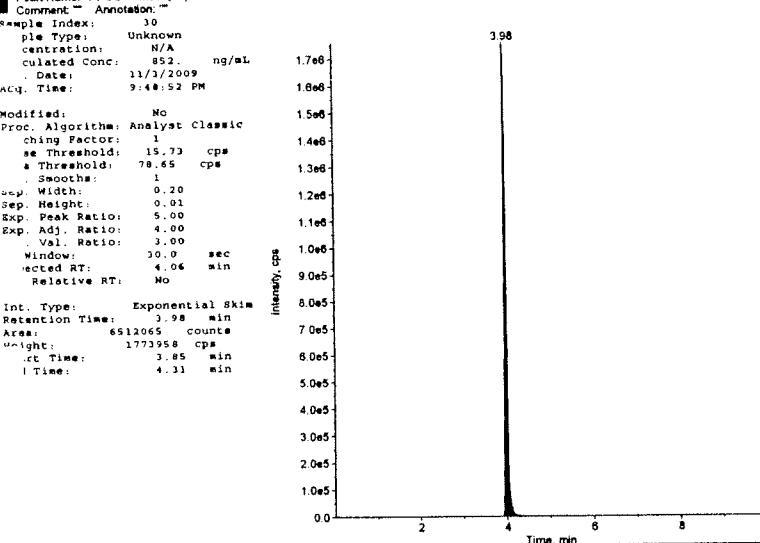
Sample Name: "L19345-2" Sample ID: "Deer #7 3.5 yr male-serum" File: "110309A.wif"
Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"
Comment: " Annotation: "



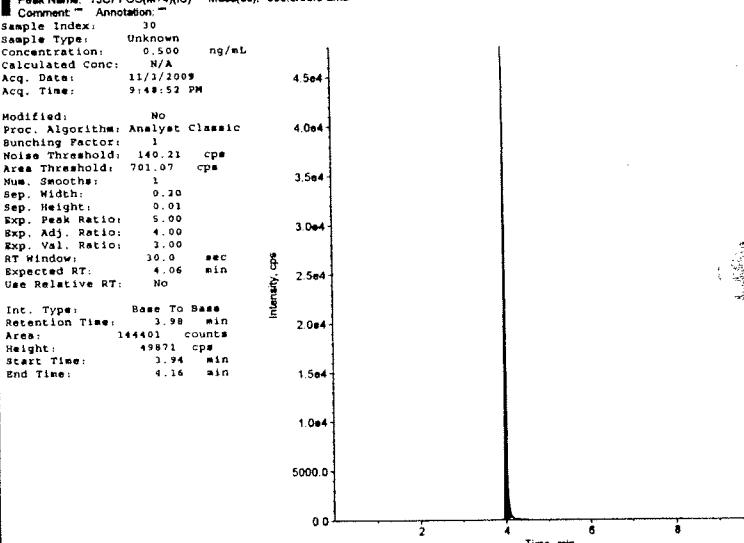
Sample Name: "L19345-2" Sample ID: "Deer #7 3.5 yr male-serum" File: "110309A.wif"
Peak Name: "13CPFOS(M+4)S" Mass(es): "503.0/80.0 amu"
Comment: " Annotation: "



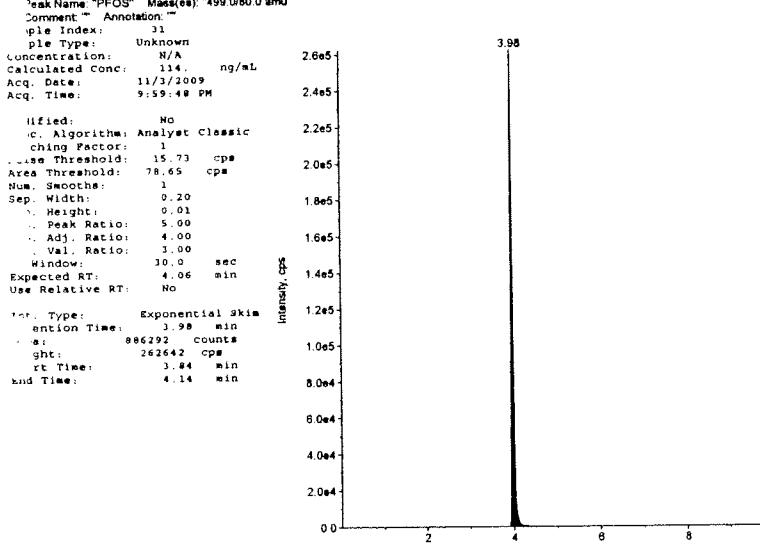
Sample Name: "L19345-2 Dup" Sample ID: "Deer #7 3.5 yr male-serum Duplicate" File: "110309A.wif"
Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"
Comment: " Annotation: "



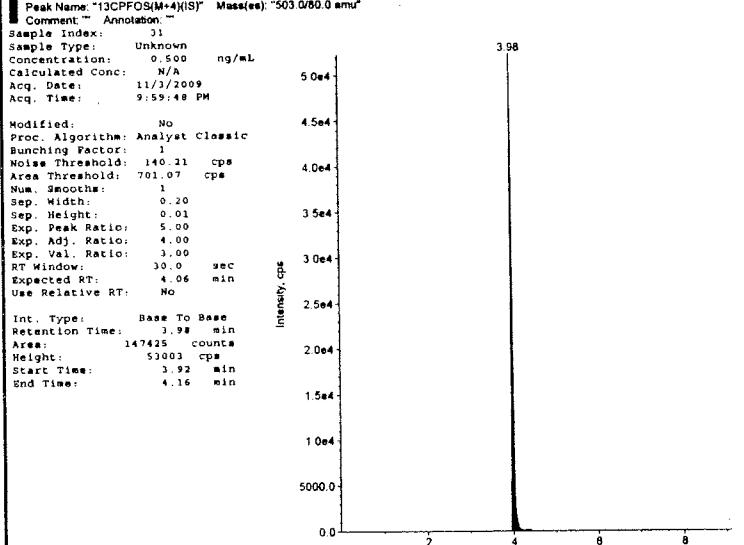
Sample Name: "L19345-2 Dup" Sample ID: "Deer #7 3.5 yr male-serum Duplicate" File: "110309A.wif"
Peak Name: "13CPFOS(M+4)S" Mass(es): "503.0/80.0 amu"
Comment: " Annotation: "



Sample Name: "L19345-1" Sample ID: "Deer #6 0.5 yr female-serum" File: "110309A.wif"
Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"
Comment: " Annotation: "



Sample Name: "L19345-1" Sample ID: "Deer #6 0.5 yr female-serum" File: "110309A.wif"
Peak Name: "13CPFOS(M+4)S" Mass(es): "503.0/80.0 amu"
Comment: " Annotation: "



lyst Version: 1.4.2
rinting Time: 8:55:47 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110309A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Sample Name: "SS33615" Sample ID: "CCV, 1.0 ng/mL" File: "110309A.wif"
Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comments: "Annotation: "

Sample Index: 32

Sample Type: QC

Concentration: 1.00 ng/mL

Calculated Conc: 1.05 ng/mL

Date: 11/3/2009

Time: 10:10:46 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Int. Type: Base To Base

Retention Time: 3.98 min

Area: 368441 counts

Height: 115045 cps

Start Time: 3.98 min

End Time: 4.26 min

3.00e4

2.00e4

1.00e4

0.00

3.98

Time, min

Sample Name: "SS33615" Sample ID: "CCV, 1.0 ng/mL" File: "110309A.wif"

Peak Name: "13CPFOS(M+4)(15)" Mass(es): "503.0/60.0 amu"

Comments: "Annotation: "

Sample Index: 32

Sample Type: QC

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/3/2009

Acq. Time: 10:10:46 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

Retention Time: 3.98 min

Area: 129728 counts

Height: 116504 cps

Start Time: 3.92 min

End Time: 4.18 min

3.00e4

2.00e4

1.00e4

0.00

3.98

Time, min

RAW DATA REPORT

Sponsor Study No: NA
 MPI Study No: L19345
 Analyte: PFOS
 Ions Monitored: 499 -> 80
 Site: NA

Limit of Quantitation: 10 ng/mL
 Injection Volume: 15 µL
 Matrix: Deer Serum

Set No: 110509A
 Analyst: Mark Neeley
 Instrument Type: LC/MS/MS Unit # 9
 Extraction Date: 11/03/05/09
 Analyzed on: 11/05/09

| MPI Research ID | Sponsor ID | Sample Code | Sample Index No. | Internal | | | | Internal | | Amount | | |
|-----------------|--------------------------------------|-------------|------------------|--------------------|--------------------|---------------------|----------------------|-----------|--------------------|-----------------------|-----------------------|--------------|
| | | | | Std. Conc. (ng/mL) | Std. Conc. (ng/mL) | Aliquot Factor (AF) | Dilution Factor (DF) | Peak Area | Standard Peak Area | Analyte Found (ng/mL) | Analyte Added (ng/mL) | Recovery (%) |
| SS33618 | - | CS | 1 | 0.100 | 1.0 | - | - | 37967 | 327142 | - | - | - |
| SS33617 | - | CS | 2 | 0.200 | 1.0 | - | - | 73226 | 332455 | - | - | - |
| SS33616 | - | CS | 3 | 0.500 | 1.0 | - | - | 178604 | 319945 | - | - | - |
| SS33615 | - | CS | 4 | 1.00 | 1.0 | - | - | 364800 | 328714 | - | - | - |
| SS33614 | - | CS | 5 | 2.00 | 1.0 | - | - | 701769 | 321742 | - | - | - |
| SS33613 | - | CS | 6 | 5.00 | 1.0 | - | - | 1710276 | 315704 | - | - | - |
| Methanol Wash | - | W | 7 | - | - | - | - | 0 | 0 | - | - | - |
| Methanol Wash | - | W | 8 | - | - | - | - | 0 | 0 | - | - | - |
| Control | MC4311 Deer Serum Control | C | 9 | - | 0.5 | 40 | 1 | 1278 | 78792 | ND | - | - |
| LCS A | MC4311 Deer Serum Spike A | LCS | 10 | - | 0.5 | 40 | 1 | 45665 | 78483 | 10.5 | 10 | 105 |
| LCS B | MC4311 Deer Serum Spike B | LCS | 11 | - | 0.5 | 40 | 1 | 242262 | 90148 | 49.2 | 50 | 98 |
| L19345-2 Spk C | Deer # 7 3.5 yr male-serum Spike C | LF | 12 | - | 0.5 | 40 | 50 | 240880 | 162940 | 1349 | 1000 | 68 |
| L19345-2 | Deer # 7 3.5 yr male-serum | S | 13 | - | 0.5 | 40 | 50 | 117915 | 159785 | 670 | - | - |
| L19345-2 Dup | Deer # 7 3.5 yr male-serum Duplicate | S | 14 | - | 0.5 | 40 | 50 | 120178 | 162619 | 671 | - | - |
| SS33615 | - | CCV | 15 | - | 1.0 | - | - | 374895 | 336883 | 1.01 | - | - |

$$\text{Analyte Found (ng/mL)} = (((\text{analyte peak area}/\text{IS peak area}) - \text{intercept}) / \text{slope}) \times \text{IS conc.} \times \text{AF} \times \text{DF}$$

Standard Curve: Linear (1/x weighted)

$$\text{Intercept} = 0.00786$$

$$\text{Slope} = 1.09$$

$$\text{Coef. Of Det.} = 1.000$$

$$\text{Recovery (\%)} = \frac{[\text{Analyte found (ng/mL)} - \text{Analyte found in control (ng/mL)}]}{\text{amount Analyte added (ng/mL)}} \times 100$$

CS = Calibration standard

LF = Lab fortified sample

W = Methanol Wash

CCV = Continuing Calibration Verification

FF = Field fortified sample

ND = Not detected = Response between 0 and LOD

C = Control sample

LCS = Laboratory Control Spike

NQ = Not quantifiable = Response between LOD and LOQ

S = Sample

Spreadsheet prepared by: Mark / 11-6-09



3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

Internal Chain of Custody/Fortification Sheet

MPI Research Study Number:
Sponsor Study/Protocol No:

L19345
NA

Matrix: Deer Serum

The samples listed below were removed from Freezer No. 46

Time 7:51 AM

Date 11-5-09

Initials mrd

| CLIENT SAMPLE ID | MPI RESEARCH ID NUMBER | VOLUME (mL) | FORTIFICATION (ng) |
|---------------------------|------------------------|-------------|--------------------|
| na | MC4311 Control | 1.0 | - |
| na | MC4311 LCS A | 1.0 | 10 |
| na | MC4311 LCS B | 1.0 | 50 |
| Deer #7 3.5 yr male-serum | L19345-2 Spk C | 1.0 | 1000 |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
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| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |
| - | - | - | - |

| | Spiking Solution Used | Volume Used for Spiking | Initial/Date |
|----------------|--------------------------|--------------------------------|--------------------|
| MC4311 LCS A | SS0033620 (200 ng/mL) | 50 μL (50-250μL auto-pipette) | <u>mrd 11-5-09</u> |
| MC4311 LCS B | SS0033619 (1000 ng/mL) | 50 μL (50-250μL auto-pipette) | <u>mrd 11-5-09</u> |
| L19345-2 Spk C | SS0032983 (100000 ng/mL) | 10 μL (0.5-10μL auto-pipette) | <u>mrd 11-5-09</u> |
| All Samples | SS0032652 (100 ng/mL) | 200 μL (50-250μL auto-pipette) | <u>mrd 11-5-09</u> |

All samples were measured using a digital autopipet IN 6914.

Time 10:00 AM

Date 11-5-09

Initials mrd

After measuring samples were returned to Freezer No. 36

Time 10:27 AM

Date 11-5-09

Initials mrd

Comments:

Analysis Summary:

Data Set: 110507A
Data Set: -
Data Set: -

Initials/Date: mrd 11-5-09

Initials/Date: - / -

Initials/Date: - / -

Set extraction/analysis data verified by: AMB

Date: 11/09/09

July 02, 2007/2



3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

SAMPLE EXTRACTION AND ANALYSIS TRACKING SHEET

MPI STUDY NUMBER: L19345
MATRIX: Deer Serum

METHOD: V5821
ANALYTICS: PFOA & PFOS

PROTOCOL NUMBER: NA

| Client ID | MPI Research ID | STEP 1 | STEP 2 | STEP 3 | STEP 4 | STEP 5 | STEP 6 | Dilutions (mL/mL) | STEP 7 | Dilutions (mL/mL) | STEP 8 | Reagents/ Materials | ExyLink ID |
|---------------------------|-----------------|--------|--------|--------|--------|--------|--------|-------------------|--------|-------------------|--------|---------------------|------------|
| na | MC4311 Control | - | - | - | - | - | - | - | - | - | - | Acetonitrile | RE47377 |
| na | MC4311 LCS A | - | - | - | - | - | - | - | - | - | - | Water | RE47376 |
| na | MC4311 LCS B | - | - | - | - | - | - | - | - | - | - | Methanol | RE47375 |
| Deer #7 3.5 yr male serum | L19345-2 Spk C | - | - | - | - | - | - | 0.02/1.0 | - | - | - | SPE Column | RE47374 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47373 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47372 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47371 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47370 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47369 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47368 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47367 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47366 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47365 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47364 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47363 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47362 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47361 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47360 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47359 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47358 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47357 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47356 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47355 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47354 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47353 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47352 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47351 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47350 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47349 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47348 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47347 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47346 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47345 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47344 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47343 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47342 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47341 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47340 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47339 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47338 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47337 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47336 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47335 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47334 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47333 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47332 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47331 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47330 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47329 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47328 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47327 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47326 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47325 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47324 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47323 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47322 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47321 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47320 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47319 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47318 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47317 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47316 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47315 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47314 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47313 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47312 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47311 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47310 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47309 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47308 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47307 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47306 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47305 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47304 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47303 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47302 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47301 |
| - | - | - | - | - | - | - | - | - | - | - | - | Initials/Date | RE47300 |

STEP 1: Measure 1 mL of serum into a 50 mL disposable centrifuge tube and fortify if appropriate. Add 0.2 mL of internal standard (100 ng/mL).

STEP 2: Add water to the sample for a final volume of 20 mL. Cap tightly and vortex for ~1 minute.

STEP 3: Transfer 1.0 mL of the sample to a 15 mL centrifuge tube. Add 5 mL of ACN and shake for ~20 minutes on a wrist action shaker.

STEP 4: Centrifuge tubes at ~3000 for ~5 minutes. Carefully decant supernatant into a 50 mL disposable centrifuge tube and add 35 mL of water.

STEP 5: SPE Cleanup: Condition column with ~10 mL of methanol followed by ~5 mL of water at a rate of ~1 drop/second. Discard all washes. Do not allow column to go dry.

STEP 6: Load the sample onto the SPE Column. Discard the eluate. Elute with 2 mL of methanol into a 15 mL centrifuge tube. Transfer the extract into autosampler vials.

STEP 7: LC/MS/MS analysis.

STEP 8: LC/MS/MS reanalysis.

*Initials and date under each step indicates the personnel that performed this step.

COMMENTS:

Final extracts stored in refrigerator 37 Initials: MM Date: 11-5-09

Oct 30, 2009/1

lyst Version: 1.4.2
rinting Time: 2:02:53 PM
rinting Date: Thursday, November 05, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MDR 11-5-09

object: \\sc1wp5556\mdrive\PE SCIEX DATA\Projects\P5195 Batch:09_110509A Serum Tab:Sample Set:SET1 AcqMethod:P5195_102

Sam

| Sample Name | Sample ID | Vial Position | Data File |
|----------------|---|---------------|--------------------|
| SS33618 | Calibration Standard, 0.1 ng/mL | 1 | 09_110509A\110509A |
| SS33617 | Calibration Standard, 0.2 ng/mL | 2 | 09_110509A\110509A |
| SS33616 | Calibration Standard, 0.5 ng/mL | 3 | 09_110509A\110509A |
| SS33615 | Calibration Standard, 1.0 ng/mL | 4 | 09_110509A\110509A |
| SS33614 | Calibration Standard, 2.0 ng/mL | 5 | 09_110509A\110509A |
| SS33613 | Calibration Standard, 5.0 ng/mL | 6 | 09_110509A\110509A |
| Methanol Wash | Methanol Wash | 91 | 09_110509A\110509A |
| Methanol Wash | Methanol Wash | 91 | 09_110509A\110509A |
| Control | MC4311 Deer Serum Control | 41 | 09_110509A\110509A |
| LCS A | MC4311 Deer Serum Spike A, 10 ng/mL | 42 | 09_110509A\110509A |
| LCS B | MC4311 Deer Serum Spike B, 50 ng/mL | 43 | 09_110509A\110509A |
| L19345-2 Spk C | Deer # 7 3.5 yr male-serum Spike C, 1000 ng/mL, DF=50 | 44 | 09_110509A\110509A |
| L19345-2 | Deer # 7 3.5 yr male-serum, DF=50 | 45 | 09_110509A\110509A |
| L19345-2 Dup | Deer # 7 3.5 yr male-serum Duplicate, DF=50 | 46 | 09_110509A\110509A |
| SS33615 | CCV, 1.0 ng/mL | 4 | 09_110509A\110509A |

LC/MS/MS SYSTEM AND OPERATING CONDITIONS

Protocol No: NA

MPI Study No: L19345

Instrument: AB API 4000 Biomolecular Mass Analyzer, (LC/MS/MS #9)
SCIEX Turbo Ion Spray Liquid Introduction Interface
Turbo Ion spray temperature = 450 °C

Computer: Dell OptiPlex GX 110

Software: PE Sciex Analyst 1.4

HPLC Equipment: Hewlett Packard (HP) Series 1100
HP Quat Pump HP Vacuum Degasser
HP Autosampler HP Column Oven

HPLC Column: Phenomenex Luna C8 (2) Mercury, 2cm x 4mm, 3 µm (ExyLIMS ID: MA0052622)

Column Temperature: 35°C

Mobile Phase (A): 2 mM Ammonium Acetate in Water (ExyLIMS ID: SL0045925)

Mobile Phase (B): Methanol (ExyLIMS ID: RE0047880)

Injected Volume: 15 µL

| <u>Time (min)</u> | <u>% A</u> | <u>% B</u> | <u>Flow Rate (µL/min)</u> |
|-------------------|------------|------------|---------------------------|
| 0.0 | 90 | 10 | 750 |
| 0.5 | 90 | 10 | 750 |
| 2.0 | 10 | 90 | 750 |
| 5.0 | 10 | 90 | 750 |
| 5.1 | 0 | 100 | 750 |
| 6.0 | 0 | 100 | 750 |
| 6.1 | 90 | 10 | 750 |
| 10.0 | 90 | 10 | 750 |

Ions monitored:

| <u>Analyte</u> | <u>Parent ion</u> | <u>Daughter ion(s)</u> | <u>Dwell (secs)</u> |
|----------------------------|-------------------|------------------------|---------------------|
| PFOA | 413 | 369 | 0.200 |
| PFOS | 499 | 80 | 0.200 |
| ¹³ C PFOA (m+2) | 415 | 370 | 0.200 |
| Internal Standard | | | |
| ¹³ C PFOS (m+4) | 503 | 80 | 0.200 |
| Internal Standard | | | |

Analyst: Mark Neeley *mnr 11-5-09*
MPI Research, Inc.
3058 Research Drive, State College, PA 16801
Phone: (814) 272-1039 FAX: (814) 231-1580

All Handwritten Peak ID's by: *mnr 11-6-09*

lyst Version: 1.4.2
rinting Time: 2:03:29 PM
rinting Date: Thursday, November 05, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MD 11-509

CXF -6.00 -6.00

Parameter Table (Period 1 Experiment 1):

AD: 7.00
CUR: 20.00
GS1: 50.00
S2: 40.00
S: -4500.00
TEM: 450.00
The: ON
E: -10.00

Agilent LC Pump Method Properties

Pump Model: Agilent 1100 LC Quaternary Pump

Minimum Pressure (psi): 0.0

Maximum Pressure (psi): 5801.0

Compressibility: 100.0

Dead Volume (µl): 40.0

Stroke Volume (µl): -1.0

Maximum Flow Ramp (ml/min²): 100.0

Maximum Pressure Ramp (psi/sec): 290.0

Step Table:

| Step | Total Time (min) | Flow Rate (µl/min) | A (%) | B (%) | C (%) | D (%) | TE#1 | TE#2 | TE#3 | TE#4 |
|------|------------------|--------------------|-------|-------|-------|-------|------|------|------|------|
| 0 | 0.00 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| | 0.50 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| | 2.00 | 750 | 0.0 | 0.0 | 10.0 | 90.0 | open | open | open | open |
| 3 | 5.00 | 750 | 0.0 | 0.0 | 10.0 | 90.0 | open | open | open | open |
| 4 | 5.10 | 750 | 0.0 | 0.0 | 0.0 | 100.0 | open | open | open | open |
| | 6.00 | 750 | 0.0 | 0.0 | 0.0 | 100.0 | open | open | open | open |
| | 6.10 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| 7 | 10.00 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |

Primary Flow Rate (µl/min): 200.0

Flow Sensor Calibration Table Index: 0

Agilent Column Oven Properties

Left Temperature (°C): 35.00

Right Temperature (°C): 35.00

Temperature Tolerance +/- (°C): 1.00

Start Acquisition Tolerance +/- (°C): 0.50

Time Table (Not Used)

Column Switching Valve Installed

Position for first sample in the batch: Left

same position for all samples in the batch

Agilent Autosampler Properties

Autosampler Model: Agilent 1100 Wellplate Autosampler

Rinse Size (µl): 100

Injection Volume (µl): 15.00

Draw Speed (µl/min): 200.0

Inject Speed (µl/min): 200.0

Sedle Level (mm): 0.00

Temperature Control Enabled

Setpoint (4 - 40 C): 4

Push Location: Flush Port

Push Time (1 - 999 sec): 10

Lc Delay Volume Reduction

Not Used

Research, Inc.

lyst Version: 1.4.2
rinting Time: 2:03:29 PM
rinting Date: Thursday, November 05, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Nov 11-5-09

Equilibration Time (sec): 2
Enable Vial/Well Bottom Sensing No
se Custom Injector Program No

lyst Version: 1.4.2
rinting Time: 2:03:29 PM
rinting Date: Thursday, November 05, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9
Nov 11-5-09

Acquisition Information:

quisition Method: P5195_102909.dam
reated: Thursday October 29 2009 09: 35: 12 AM
Last Modified: Friday October 30 2009 10: 56: 20 AM
Comment:
ynchronization Mode: PFOA PFOS
uto-Equilibration: LC Sync
Acquisition Duration: Off
Number Of Scans: 10min0sec
eriods In File: 732
Acquisition Module: 1
Software version: Acquisition Method
Analyst 1.4.2

Period 1:

Scans in Period: 732
relative Start Time: 0.00 msec
Experiments in Period: 1

Period 1 Experiment 1:

Scan Type: MRM (MRM)
Polarity: Negative
can Mode: N/A
on Source: Turbo Spray
Resolution Q1: Unit
Resolution Q3: Unit
n. Intensity Thres.: 0.00 cps
etting Time: 0.0000 msec
MR Pause: 5.0070 msec
DA: No
Step Size: 0.00 amu

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 13.00 | 369.00 | 200.00 | DF | -32.00 | -32.00 |
| | | | CE | -18.00 | -18.00 |
| | | | CXE | -13.20 | -13.20 |

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 415.00 | 370.00 | 200.00 | DF | -32.00 | -32.00 |
| | | | CE | -18.00 | -18.00 |
| | | | CXE | -13.20 | -13.20 |

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 499.00 | 80.00 | 200.00 | DF | -83.00 | -83.00 |
| | | | CE | -88.00 | -88.00 |
| | | | CXE | -6.00 | -6.00 |

| Q1 Mass (amu) | Q3 Mass (amu) | Dwell (msec) | Param | Start | Stop |
|---------------|---------------|--------------|-------|--------|--------|
| 503.00 | 80.00 | 200.00 | DF | -83.00 | -83.00 |
| | | | CE | -88.00 | -88.00 |

lyst Version: 1.4.2
rinting Time: 2:03:29 PM
rinting Date: Thursday, November 05, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

MD, 11-5-09

CXE -6.00 -6.00

parameter Table(Period 1 Experiment 1):

WD: 7.00
CUR: 20.00
GS1: 50.00
S2: 40.00
S: -4500.00
TEM: 450.00
The: ON
E: -10.00

Agilent LC Pump Method Properties

Pump Model: Agilent 1100 LC Quaternary Pump
Minimum Pressure (psi): 0.0
Maximum Pressure (psi): 5801.0
Compressibility: 100.0
Dead Volume (μl): 40.0
Stroke Volume (μl): -1.0
Maximum Flow Ramp (ml/min²): 100.0
Maximum Pressure Ramp (psi/sec): 290.0

Step Table:

| Step | Total Time (min) | Flow Rate (μl/min) | A (%) | B (%) | C (%) | D (%) | TE#1 | TE#2 | TE#3 | TE#4 |
|------|------------------|--------------------|-------|-------|-------|-------|------|------|------|------|
| 0 | 0.00 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| | 0.50 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| | 2.00 | 750 | 0.0 | 0.0 | 10.0 | 90.0 | open | open | open | open |
| 3 | 5.00 | 750 | 0.0 | 0.0 | 10.0 | 90.0 | open | open | open | open |
| 4 | 5.10 | 750 | 0.0 | 0.0 | 0.0 | 100.0 | open | open | open | open |
| | 6.00 | 750 | 0.0 | 0.0 | 0.0 | 100.0 | open | open | open | open |
| | 6.10 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |
| 7 | 10.00 | 750 | 0.0 | 0.0 | 90.0 | 10.0 | open | open | open | open |

Primary Flow Rate (μl/min): 200.0

Flow Sensor Calibration Table Index: 0

Agilent Column Oven Properties

Left Temperature (°C): 35.00
Right Temperature (°C): 35.00
Temperature Tolerance +/- (°C): 1.00
Start Acquisition Tolerance +/- (°C): 0.50

Time Table (Not Used)

Column Switching Valve Installed

Position for first sample in the batch: Left
same position for all samples in the batch

Agilent Autosampler Properties

Autosampler Model: Agilent 1100 Wellplate Autosampler
Vial Size (μl): 100
Injection Volume (μl): 15.00
Draw Speed (μl/min): 200.0
Inject Speed (μl/min): 200.0
Needle Level (mm): 0.00
Temperature Control Enabled
Setpoint (4 - 40 C): 4
Push Location: Flush Port
Push Time (1 - 999 sec): 10

Automatic Delay Volume Reduction Not Used

Research, Inc.

lyst Version: 1.4.2
rinting Time: 2:03:29 PM
rinting Date: Thursday, November 05, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Mon 11-5-09

Equilibration Time (sec): 2
End Vial/Well Bottom Sensing No
se Custom Injector Program No

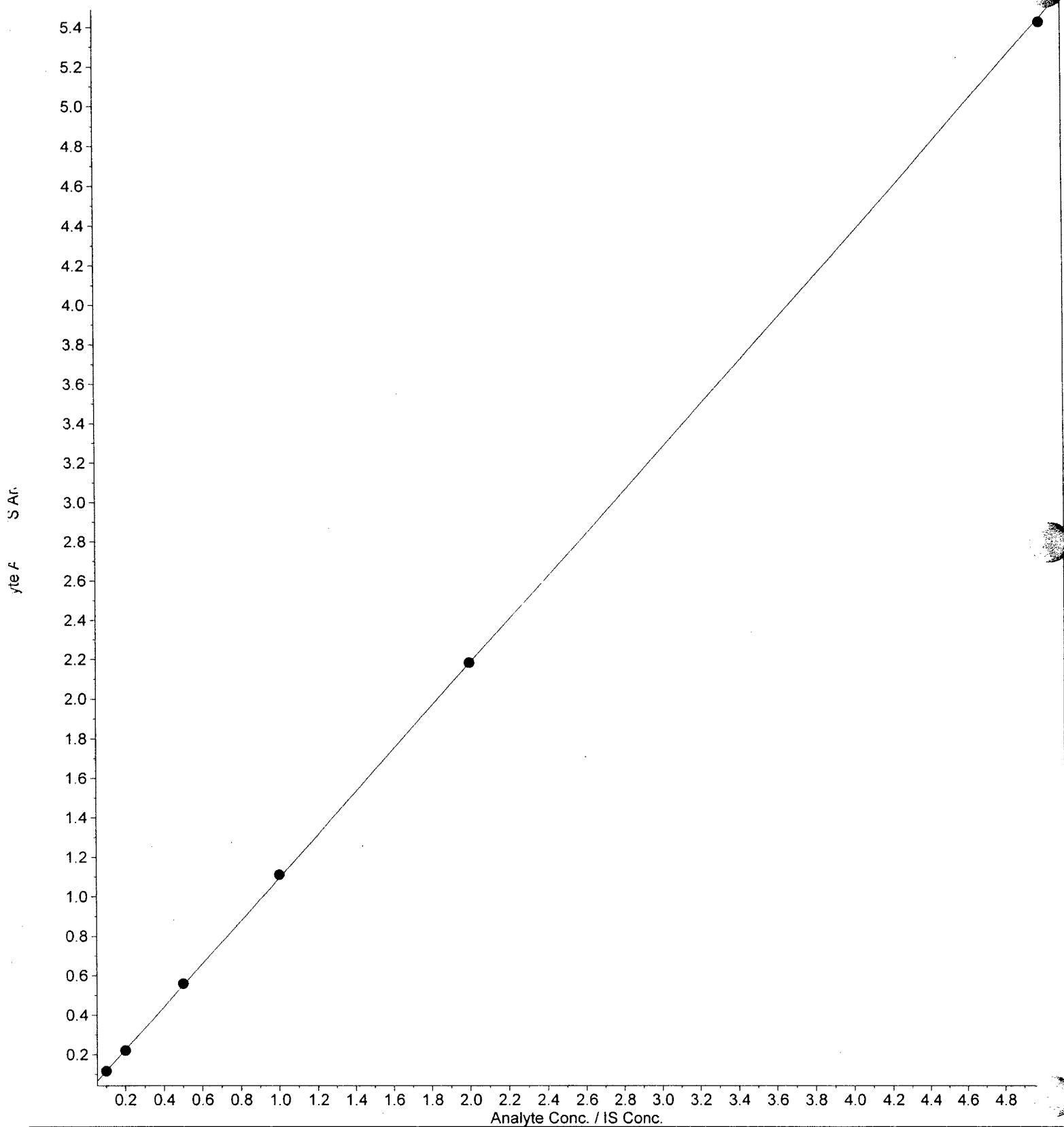
lyst Version: 1.4.2
rinting Time: 8:51:45 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

Nov 11-6-09

109_110509A.rdb (PFOS): "Linear" Regression ("1 / x" weighting): $y = 1.09 x + 0.00786$ ($r = 1.0000$)



lyst Version: 1.4.2

rinting Time: 8:52:24 AM

rinting Date: Friday, November 06, 2009

MPI Study: L19345

MPI Set No.: 110509A

Operator: Mark Neeley

Instrument No.: LC/MS/MS #9

Sample Name: "SS33618" Sample ID: "Calibration Standard, 0.1 ng/mL" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comment: "Annotation: "

Sample Index: 1

Sample Type: Standard

Concentration: 0.100 ng/mL

3.99

Calculated Conc: 0.0996 ng/mL

Date: 11/5/2009

Time: 5:36:33 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 9500.00

Noise Threshold: 15.73 cps

Smooths: 1

Width: 0.20

Height: 0.01

Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Type: Base To Base

Retention Time: 3.99 min

Area: 37967 counts

Height: 11514 cps

Start Time: 3.90 min

End Time: 4.21 min

3000.00

2500.00

2000.00

1500.00

1000.00

500.00

0.00

Sample Name: "SS33618" Sample ID: "Calibration Standard, 0.1 ng/mL" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(IS)" Mass(es): "503.0/60.0 amu"

Comment: "Annotation: "

Sample Index: 1

Sample Type: Standard

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/5/2009

Acq. Time: 5:36:33 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 14.21 cps

Area Threshold: 701.07 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Intensity, cps

5.50e4

5.00e4

4.50e4

4.00e4

3.50e4

3.00e4

2.50e4

2.00e4

1.50e4

1.00e4

500.00

0.00

Sample Name: "SS33617" Sample ID: "Calibration Standard, 0.2 ng/mL" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comment: "Annotation: "

Sample Index: 2

Sample Type: Standard

Concentration: 0.200 ng/mL

3.99

Calculated Conc: 0.196 ng/mL

Date: 11/5/2009

Time: 5:48:37 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 70.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Type: Base To Base

Retention Time: 3.99 min

Area: 73226 counts

Height: 22270 cps

Start Time: 3.88 min

End Time: 4.20 min

6000.00

5000.00

4000.00

3000.00

2000.00

1000.00

0.00

Sample Name: "SS33617" Sample ID: "Calibration Standard, 0.2 ng/mL" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(IS)" Mass(es): "503.0/60.0 amu"

Comment: "Annotation: "

Sample Index: 2

Sample Type: Standard

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/5/2009

Acq. Time: 5:48:37 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 14.21 cps

Area Threshold: 701.07 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Intensity, cps

5.50e4

5.00e4

4.50e4

4.00e4

3.50e4

3.00e4

2.50e4

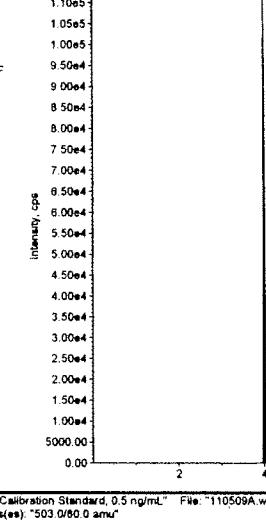
2.00e4

1.50e4

1.00e4

500.00

0.00



Sample Name: "SS33616" Sample ID: "Calibration Standard, 0.5 ng/mL" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/60.0 amu"

Comment: "Annotation: "

Sample Index: 3

Sample Type: Standard

Concentration: 0.500 ng/mL

3.99

Calculated Conc: 0.507 ng/mL

Acq. Date: 11/5/2009

Acq. Time: 5:59:34 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 4.5e4

Noise Threshold: 15.73 cps

Area Threshold: 70.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Type: Base To Base

Retention Time: 3.99 min

a: 178604 counts

b: 55621 cps

rt Time: 3.90 min

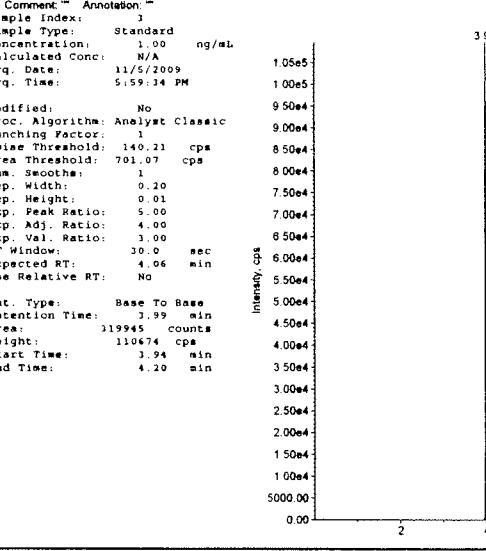
End Time: 4.24 min

1.5e4

1.0e4

5000.00

0.00



Research, Inc.

Page 1 of 5

Initials mon

Date 11-6-09

Sample

Index /

To 15

lyst Version: 1.4.2

rinting Time: 8:52:25 AM

MPI Study: L19345

MPI Set No.: 110509A

Operator: Mark Neeley

Instrument No.: LC/MS/MS #9

rinting Date: Friday, November 06, 2009

Sample Name: "SS33615" Sample ID: "Calibration Standard, 1.0 ng/mL" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 4

Sample Type: Standard

Concentration: 1.00 ng/mL 1.05e5

Calculated Conc: 1.01 ng/mL 8.50e4

Acq. Date: 11/5/2009 1.00e5

Acq. Time: 6:10:31 PM 9.50e4

Modified: No 9.00e4

Proc. Algorithm: Analyst Classic 9.00e4

Bunching Factor: 1 9.00e4

Noise Threshold: 15.73 cps 8.50e4

Area Threshold: 78.65 cps 8.00e4

Smooths: 1 8.00e4

Sep. Width: 0.20 7.50e4

Sep. Height: 0.01 7.00e4

Peak Ratio: 5.00 7.00e4

Exp. Adj. Ratio: 4.00 6.50e4

Exp. Val. Ratio: 3.00 6.50e4

RT Window: 30.0 sec 6.00e4

Expected RT: 4.06 min 6.00e4

Relative RT: No 5.50e4

Type: Base To Base 5.00e4

Retention Time: 4.00 min 4.50e4

Area: 364800 counts 4.00e4

Height: 108831 cps 4.00e4

Start Time: 3.87 min 3.50e4

End Time: 4.22 min 3.00e4

Intensity, cps 3.00e4

2.50e4 2.00e4

1.50e4 1.00e4

5000.00 5000.00

0.00 0.00

4.00

Time, min

Sample Name: "SS33615" Sample ID: "Calibration Standard, 1.0 ng/mL" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)IS" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 4

Sample Type: Standard

Concentration: 1.00 ng/mL 1.10e5

Calculated Conc: N/A 1.10e5

Acq. Date: 11/5/2009 1.05e5

Acq. Time: 6:10:31 PM 1.05e5

Modified: No 1.00e5

Proc. Algorithm: Analyst Classic 9.50e4

Bunching Factor: 1 9.00e4

Noise Threshold: 140.21 cps 8.50e4

Area Threshold: 701.07 cps 8.00e4

Num. Smooths: 1 8.00e4

Sep. Width: 0.20 8.00e4

Sep. Height: 0.01 7.50e4

Peak Ratio: 5.00 7.00e4

Exp. Adj. Ratio: 4.00 7.00e4

Exp. Val. Ratio: 3.00 6.50e4

RT Window: 30.0 sec 6.50e4

Expected RT: 4.06 min 6.00e4

Use Relative RT: No 5.50e4

Intensity, cps 5.50e4

5.00e4 4.50e4

4.00e4 3.50e4

3.00e4 2.50e4

2.00e4 1.50e4

1.00e4 5000.00

0.00 5000.00

3.99

Time, min

Sample Name: "SS33814" Sample ID: "Calibration Standard, 2.0 ng/mL" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 5

Sample Type: Standard

Concentration: 2.00 ng/mL 2.1e5

Calculated Conc: 2.00 ng/mL 2.0e5

Acq. Date: 11/5/2009 2.0e5

Acq. Time: 6:21:29 PM 1.9e5

Modified: No 1.8e5

Proc. Algorithm: Analyst Classic 1.7e5

Bunching Factor: 1 1.7e5

Noise Threshold: 15.73 cps 1.6e5

Area Threshold: 78.65 cps 1.6e5

Smooths: 1 1.5e5

Sep. Width: 0.20 1.5e5

Sep. Height: 0.01 1.4e5

Peak Ratio: 5.00 1.3e5

Exp. Adj. Ratio: 4.00 1.3e5

Exp. Val. Ratio: 3.00 1.2e5

RT Window: 30.0 sec 1.2e5

Expected RT: 4.06 min 1.1e5

Relative RT: No 1.1e5

Intensity, cps 1.0e5

9.0e4 8.0e4

7.0e4 6.0e4

5.0e4 4.0e4

3.0e4 2.0e4

1.0e4 5000.00

0.00 5000.00

Time, min

3.99

Time, min

Sample Name: "SS33814" Sample ID: "Calibration Standard, 2.0 ng/mL" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)IS" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 5

Sample Type: Standard

Concentration: 1.00 ng/mL 1.10e5

Calculated Conc: N/A 1.05e5

Acq. Date: 11/5/2009 1.05e5

Acq. Time: 6:21:29 PM 1.00e5

Modified: No 9.50e4

Proc. Algorithm: Analyst Classic 9.00e4

Bunching Factor: 1 9.00e4

Noise Threshold: 140.21 cps 8.50e4

Area Threshold: 701.07 cps 8.00e4

Num. Smooths: 1 8.00e4

Sep. Width: 0.20 8.00e4

Sep. Height: 0.01 7.50e4

Peak Ratio: 5.00 7.00e4

Exp. Adj. Ratio: 4.00 7.00e4

Exp. Val. Ratio: 3.00 6.50e4

RT Window: 30.0 sec 6.50e4

Expected RT: 4.06 min 6.00e4

Use Relative RT: No 5.50e4

Intensity, cps 5.50e4

5.00e4 4.50e4

4.00e4 3.50e4

3.00e4 2.50e4

2.00e4 1.50e4

1.00e4 5000.00

0.00 5000.00

Time, min

3.99

Time, min

lyst Version: 1.4.2
rinting Time: 8:52:25 AM
rinting Date: Friday, November 06, 2009

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

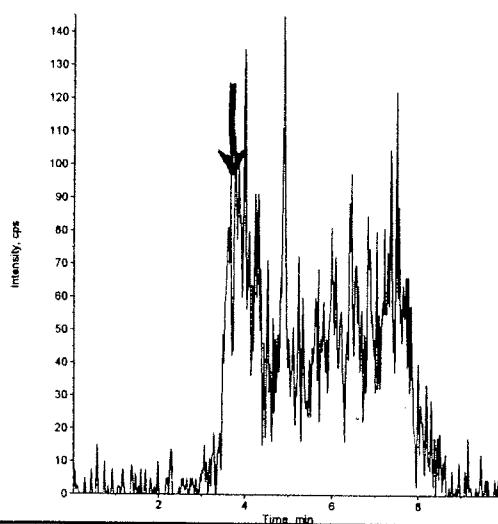
Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 7
Sample Type: Solvent
Concentration: 0.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/5/2009
Acq. Time: 6:43:25 PM

Modified: Yes

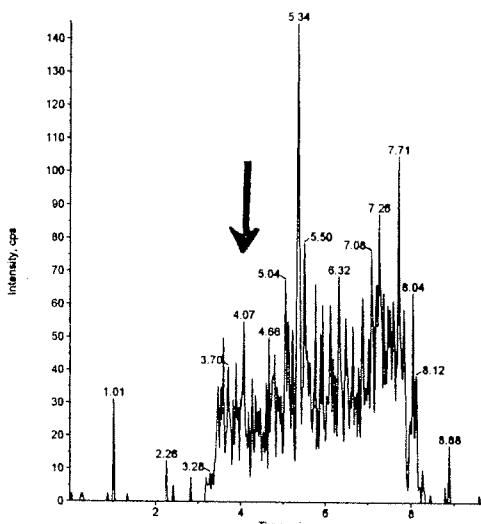


Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110509A.wif"
Peak Name: "13CPFOS(M+4)(IS)" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 7
Sample Type: Solvent
Concentration: 1.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/5/2009
Acq. Time: 6:43:25 PM

Modified: Yes



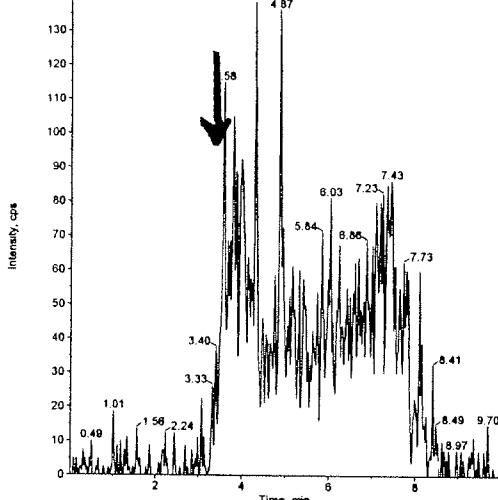
Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 8
Sample Type: Solvent
Concentration: 0.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/5/2009
Acq. Time: 6:54:22 PM

Modified: Yes



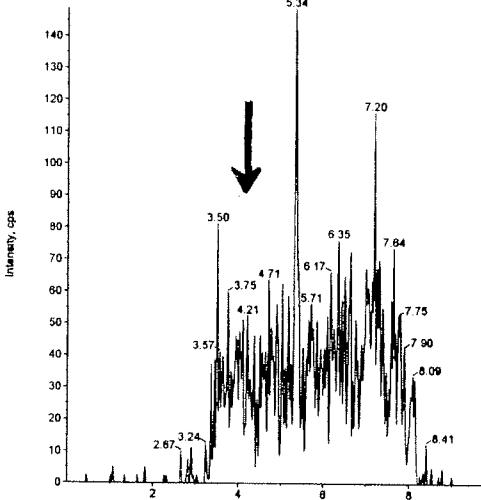
Sample Name: "Methanol Wash" Sample ID: "Methanol Wash" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(IS)" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 8
Sample Type: Solvent
Concentration: 1.00 ng/mL
Calculated Conc: N/A
Acq. Date: 11/5/2009
Acq. Time: 6:54:22 PM

Modified: Yes



Sample Name: "Control" Sample ID: "MC4311 Deer Serum Control" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 9
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0771 ng/mL
Acq. Date: 11/5/2009
Acq. Time: 7:05:18 PM

Modified: No

Proc. Algorithm: Analyst Classic
Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 70.65 cps

Num. Smooths: 1

Imp. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

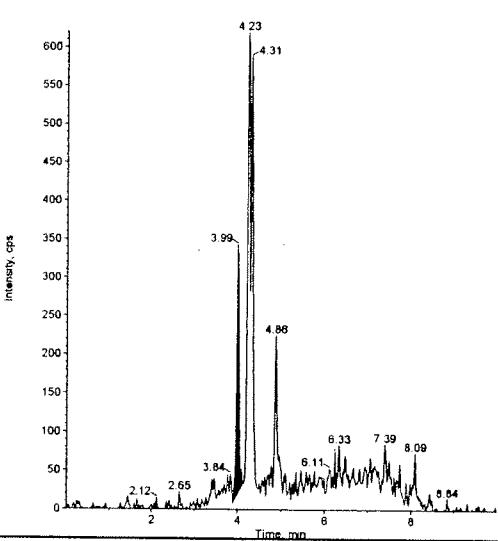
Retention Time: 3.99 min

A: 1278 counts

Height: 324 cps

Start Time: 3.94 min

End Time: 4.14 min



Sample Name: "Control" Sample ID: "MC4311 Deer Serum Control" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(IS)" Mass(es): "503.0/80.0 amu"

Comment: " Annotation: "

Sample Index: 9
Sample Type: Unknown
Concentration: 0.350 ng/mL
Calculated Conc: N/A
Acq. Date: 11/5/2009
Acq. Time: 7:05:18 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

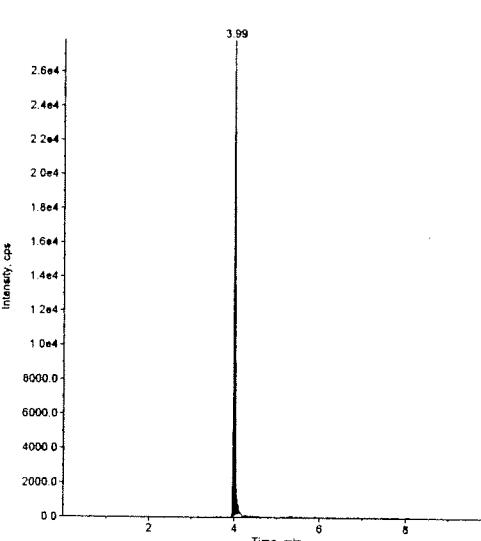
Retention Time: 3.99 min

A: 78792 counts

Height: 27766 cps

Start Time: 3.94 min

End Time: 4.14 min

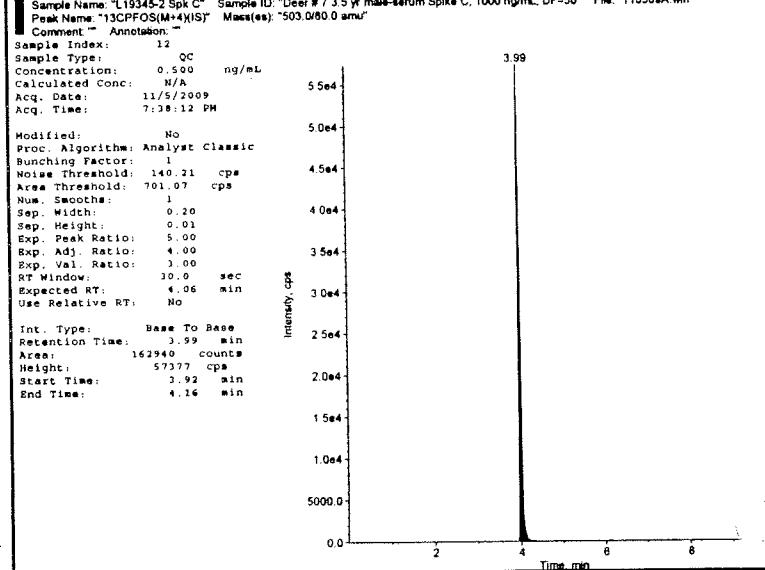
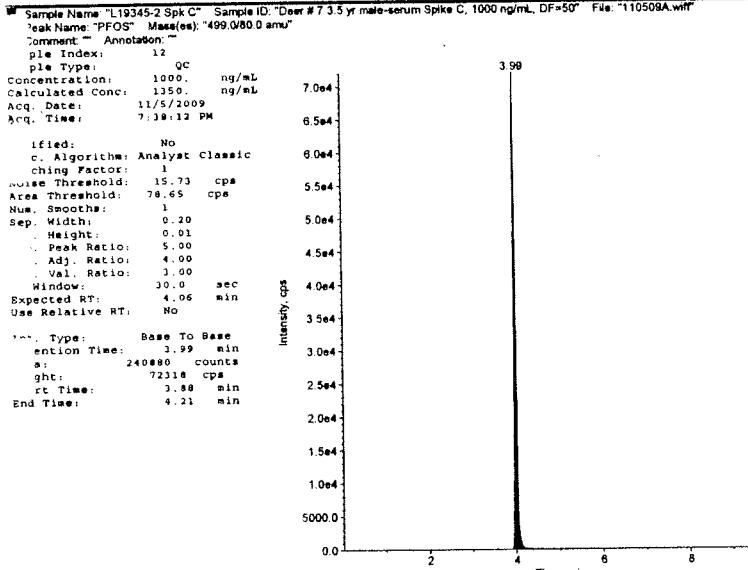
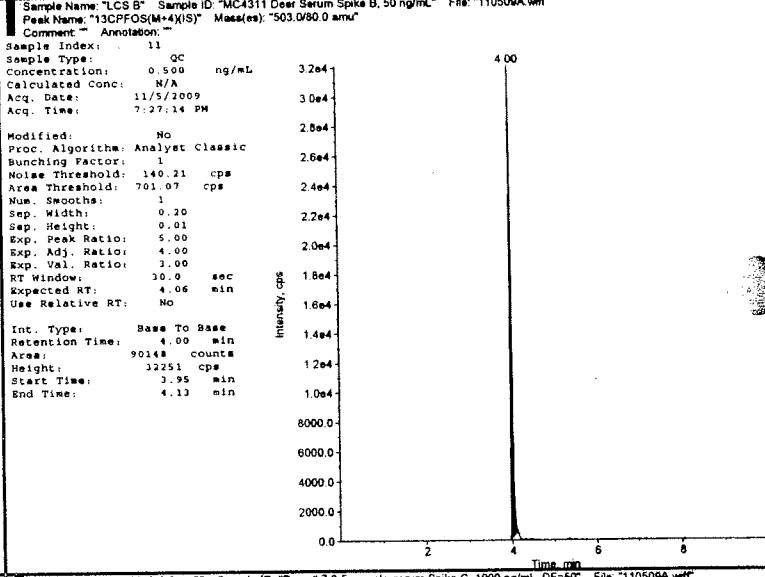
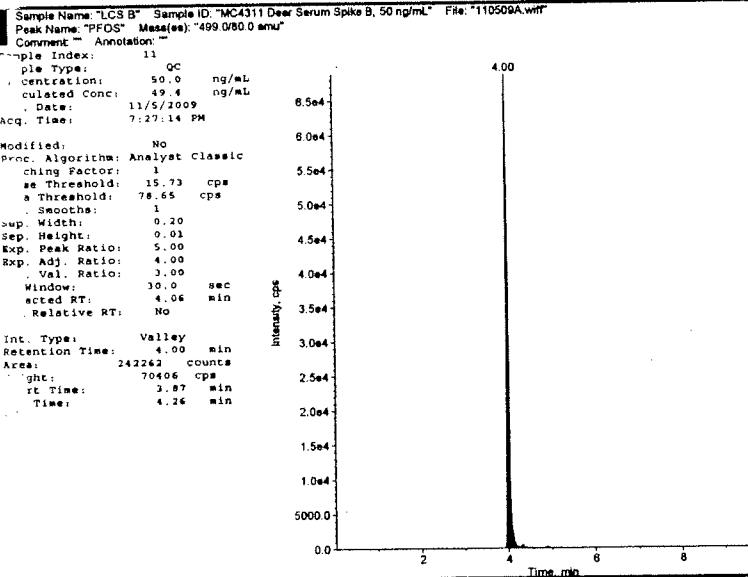
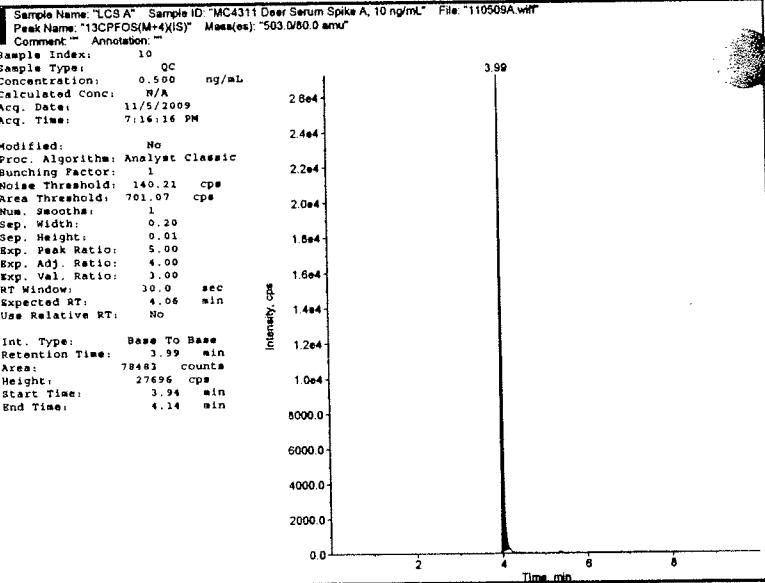
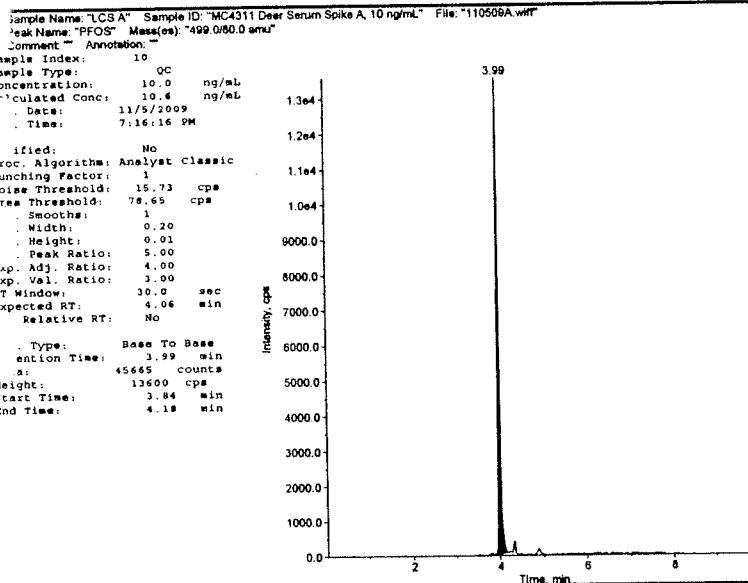


lyst Version: 1.4.2
rinting Time: 8:52:25 AM

MPI Study: L19345
MPI Set No.: 110509A

Operator: Mark Neeley
Instrument No.: LC/MS/MS #9

rinting Date: Friday, November 06, 2009



Sample Name: "L19345-2" Sample ID: "Deer #7.3.5 yr male-serum, DF=50" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 13

Sample Type: Unknown

Concentration: N/A

Calculated Conc: 672. ng/mL

Acq. Date: 11/5/2009

Acq. Time: 7:49:11 PM

ifid: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Relative RT: No

Int. Type: Base To Base

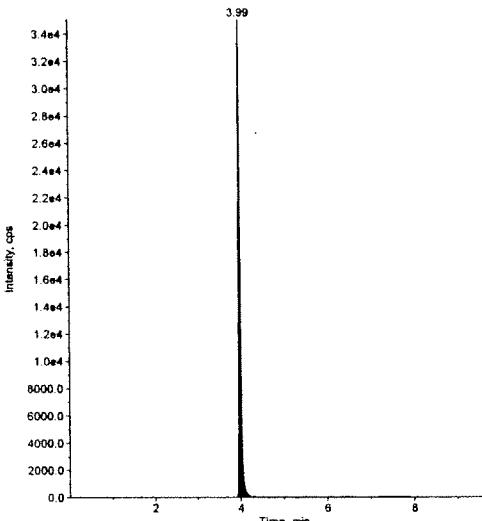
Retention Time: 3.99 min

Area: 117915 counts

Height: 35078 cps

Start Time: 3.87 min

End Time: 4.28 min



Sample Name: "L19345-2" Sample ID: "Deer #7.3.5 yr male-serum, DF=50" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(S)" Mass(es): "503.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 13

Sample Type: Unknown

Concentration: 0.500 ng/mL

Calculated Conc: N/A

Acq. Date: 11/5/2009

Acq. Time: 7:49:11 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

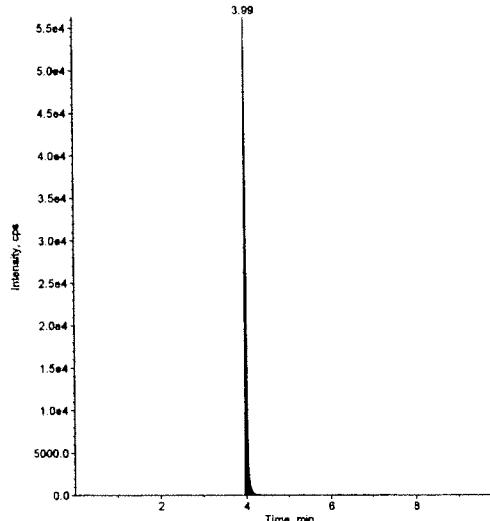
Retention Time: 3.99 min

Area: 159785 counts

Height: 56315 cps

Start Time: 3.94 min

End Time: 4.17 min



Sample Name: "L19345-2 Dup" Sample ID: "Deer #7.3.5 yr male-serum Duplicate, DF=50" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 14

Sample Type: Unknown

Concentration: N/A

Calculated Conc: 673. ng/mL

Acq. Date: 11/5/2009

Acq. Time: 8:00:10 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

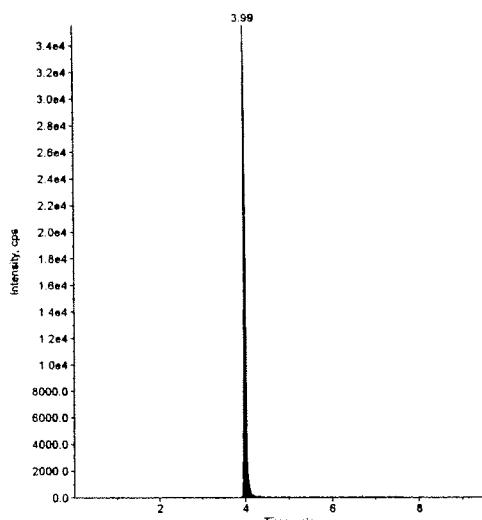
Retention Time: 3.99 min

Area: 120178 counts

Height: 35601 cps

Start Time: 3.87 min

End Time: 4.18 min



Sample Name: "L19345-2 Dup" Sample ID: "Deer #7.3.5 yr male-serum Duplicate, DF=50" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(S)" Mass(es): "503.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 14

Sample Type: Unknown

Concentration: 0.500 ng/mL

Calculated Conc: N/A

Acq. Date: 11/5/2009

Acq. Time: 8:00:10 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

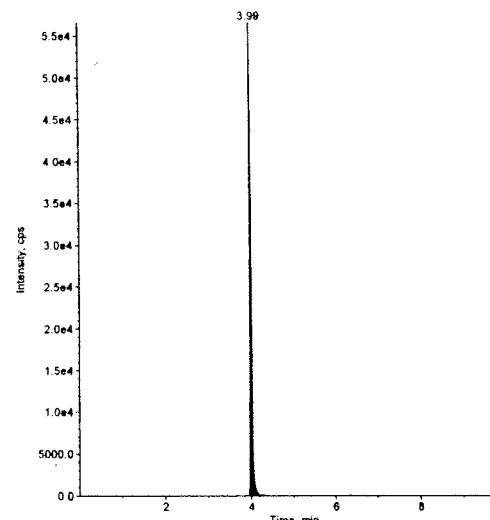
Retention Time: 3.99 min

Area: 162619 counts

Height: 56620 cps

Start Time: 3.94 min

End Time: 4.18 min



Sample Name: "SS33615" Sample ID: "CCV, 1.0 ng/mL" File: "110509A.wif"

Peak Name: "PFOS" Mass(es): "499.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 15

Sample Type: QC

Concentration: 1.00 ng/mL

Calculated Conc: 1.02 ng/mL

Acq. Date: 11/5/2009

Acq. Time: 8:11:09 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 15.73 cps

Area Threshold: 78.65 cps

Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No

Int. Type: Base To Base

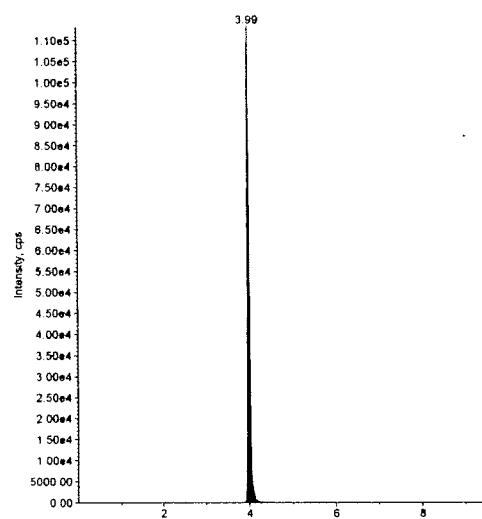
Retention Time: 3.99 min

Area: 374895 counts

Height: 114055 cps

Start Time: 3.85 min

End Time: 4.13 min



Sample Name: "SS33615" Sample ID: "CCV, 1.0 ng/mL" File: "110509A.wif"

Peak Name: "13CPFOS(M+4)(S)" Mass(es): "503.0/80.0 amu"

Comment: "Annotation: "

Sample Index: 15

Sample Type: QC

Concentration: 1.00 ng/mL

Calculated Conc: N/A

Acq. Date: 11/5/2009

Acq. Time: 8:11:09 PM

Modified: No

Proc. Algorithm: Analyst Classic

Bunching Factor: 1

Noise Threshold: 140.21 cps

Area Threshold: 701.07 cps

Num. Smooths: 1

Sep. Width: 0.20

Sep. Height: 0.01

Exp. Peak Ratio: 5.00

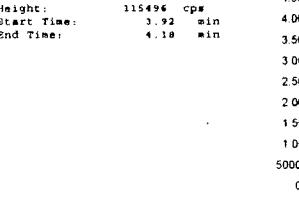
Exp. Adj. Ratio: 4.00

Exp. Val. Ratio: 3.00

RT Window: 30.0 sec

Expected RT: 4.06 min

Use Relative RT: No



BIORECLAMATION INC.
123 FROST STREET
WESTBURY, NEW YORK 11590
516-483-1196 FAX 516-483-4683
E-MAIL:techs@Bioreclamation.com

CERTIFICATE OF ANALYSIS

**PRODUCT: DEER PROJECT
CATALOG # DER-PROJECT
LOT# DERBREC.101 – DERBREC.103
STORAGE TEMPERATURE: -20^o C OR COLDER**

DATE: 10/21/09

PURCHASE ORDER: SC11-04103

PROJECT NUMBER: 1061420

PRESENTATION:

1. 100 ML SERUM mc 4311/mc 4312
LOT: DERBREC.101
2. 1 DEER LIVER mc 4313
LOT: DERBREC.102
3. 500 G DEER MUSCLE mc 4314
LOT: DERBREC.103

FILTRATION: NONE

EXPIRATION: 10/2011

BIOHAZARD INFO: THIS MATERIAL SHOULD BE HANDLED AS IF CAPABLE OF TRANSMITTING INFECTIOUS AGENTS. PLEASE USE UNIVERSAL PRECAUTIONS.

THIS PRODUCT IS BEING SOLD FOR RESEARCH AND, OR MANUFACTURING PURPOSES ONLY. IT IS NOT TO BE USED IN HUMANS OR ANIMALS. FOR IN VITRO USE ONLY. THE USER ASSUMES ALL RESPONSIBILITY FOR ITS USAGE AND DISPOSAL, IN ACCORDANCE WITH ALL REGULATIONS.

PLEASE FOLD THIS SHIPPING DOCUMENT IN HALF AND PLACE IT IN A WAYBILL POUCH AFFIXED TO YOUR SHIPMENT SO THAT THE BARCODE PORTION OF THE LABEL CAN BE READ AND SCANNED. **WARNING: USE ONLY THE PRINTED ORIGINAL LABEL FOR SHIPPING. USING A PHOTOCOPY OF THIS LABEL FOR SHIPPING PURPOSES IS FRAUDULENT AND COULD RESULT IN ADDITIONAL BILLING CHARGES, ALONG WITH THE CANCELLATION OF YOUR FEDEX ACCOUNT NUMBER.

From: Origin ID: RMEA (516) 483-1196
GLENN FRUCHTNIS
BIORECLAMATION INC.
123 FROST ST

WESTBURY, NY 11590



Ship Date: 20OCT09 Dry Ice: 0.5 KGS
ActWgt: 5.0 LB MAN
System#: 397072/CAFE2361
Account#: S 011515100

Delivery Address Bar Code



Ref #
Invoice #
PO #
Dept #

SHIP TO: (814) 231-8032 BILL SENDER
PAUL CONNOLY
MPI RESEARCH (EXYGEN)
3048 RESEARCH DRIVE
STATE COLLEGE, PA 16801

STANDARD OVERNIGHT

WED

Deliver By:
21OCT09

TRK# 9887 6350 1371 FORM 0201

PIT AA

16801 -PA-US
DSR-ICE

NA SCEA

